BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- BLURRY OR ILLEGIBLE TEXT
- SKEWED/SLATED IMAGES
- COLORED PHOTOS
- BLACK OR VERY DARK BLACK AND WHITE PHOTOS
- UNDECIPHERABLE GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

Express Mail Label No. EV443088460US

PATENT APPLICATION
Docket No. 6300.96.1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of		)
	A. Bruno Frazier et al.	)
Serial No.:	09/787,498	)
Filed:	March 16, 2001	) Art Unit 3743
Conf. No.:	3953	)
For:	SURFACE MICROMACHINED MICRONEEDLES	)
Confirmation No.:	3953	)
Examiner:	Kathryn Odland	)

DECLARATION UNDER 37 C.F.R. § 1.131

We, A. Bruno Frazier and John D. Brazzle declare as follows:

1. We are the coinventors of the invention claimed in the above-identified patent application Serial No. 09/787,498. We were previously employed by The University of Utah, in Salt Lake City, Utah.

2. During our employment at The University of Utah and prior to June 10, 1998, we conceived the idea of surface micromachined microneedle devices as described and claimed in the above-identified patent application in this country. Copies of invention disclosure documents describing and showing various inventive concepts of the presently claimed invention and their reduction to practice are attached hereto as Exhibits A and B.

3. All of the claimed embodiments of the invention were conceived prior to June 10, 1998, while some of these embodiments were reduced to practice prior to June 10, 1998, and others were reduced to practice with due diligence after June 10, 1998.

4. Exhibit A comprises laboratory notebook pages 1-42, which include written descriptions, drawings, manufacturing procedures, and photographs of various microneedle array embodiments of the invention. Each of the dates deleted from Exhibit A are prior to June 10, 1998.

5. Photomicrographs of microneedle array devices of the invention fabricated prior to June 10, 1998 are shown on pages 28, and 35-37 of Exhibit A.

6. Exhibit B comprises laboratory notebook pages 43-81, which include written descriptions, drawings, manufacturing procedures, and photographs of single microneedle embodiments of the invention.

7. Photomicrographs of single microneedle devices of the invention fabricated after June 10, 1998 are shown on pages 50, 51, 65, 66, 68, 69, 80, and 81 of Exhibit B.

8. We declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 1st day of April 2004.

Inventor: A. Bruno Frazier

Dated this 1st day of April 2004.

Inventor: _____
John D. Brazzle

John Bragg Needles Frazier 355-3076 1/12-97

Meeting w/ Frazier @ 1500 hrs on 16 Apr 97.
-possible project on a 3-Dimensional Micromachined Needle Array. Would be in charge of:
Company Requirements

Phase I: Demonstration that a two-dimensional microneedle array and manifold can actually be made.

The dimensions, etc. of the array and manifold for this phase would be: (if you think there are better dimensions, indicate where you would select different dimensions)

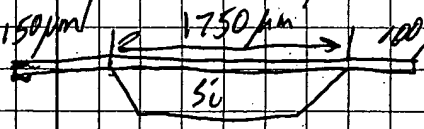
- overall array size 5mm x 5mm square
- needle spacing 200u centers--the array would be 25x25
- needle size 75u wide, less than 75u thick on the outside
  40u wide by greater than 10u on the inside
- needle length 150u from base of needle to tip of needle
- needle tip geometry 75 deg bevel on narrow dim, 45 deg on wide dim
- manifold thickness less than 3 mm
- volume of manifold 1mm x 5mm x 5mm (on the inside)
  (above needle array
  and below the luer)
- syringe interface standard luer

Deliverables: 10 devices of the above geometry, greater than 90% of needles are open per array, when placed on a syringe fluid is expressed from the manifold with roughly equal velocity (and the manifold is capable of handling the internal pressures--less than 100 PSI--without breaking).

From [redacted] Anonymous Co.

Start Date: ≥ June or sooner (awaiting contract approval)
End Date: Phase I 5or one year
personal: Attempt to finish by Christmas

Conceptual Design Ideas:



nee dle

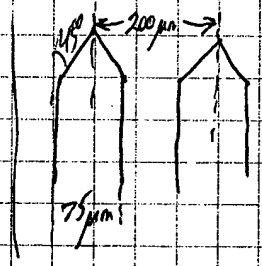
Fits 2mm dim

Thickness Consideration:
25 needles thick: w/max 75μm structure need 125μm thick Si wafer

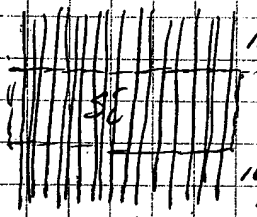
Could design structure to be around 100μm high. Use Si wafer of 6 mils = 150μm slightly beyond 5mm required

2

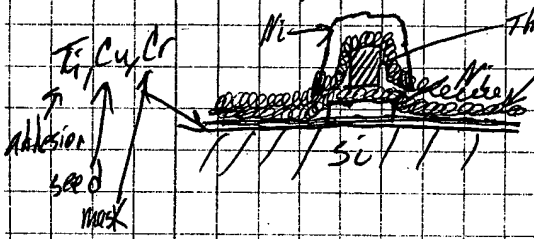
25 Needles wide



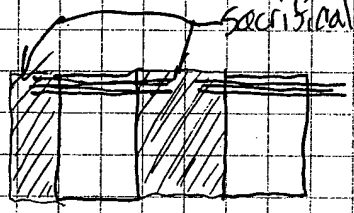
leaves 62.5 μm of Si on either side of array



will utilize existing PR procedure to form hollow rectangular channels

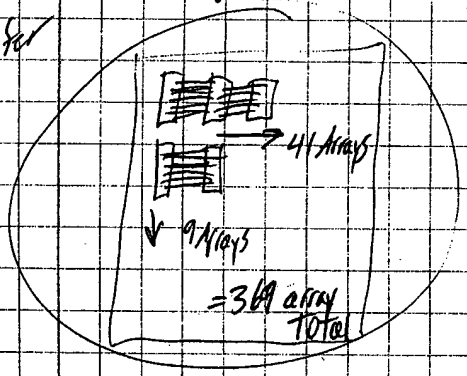


Apply PR, etch Cr, plate Ni
Sputter Au, or Ti Apply Thick PR, pattern,
Sputter Au, pattern PR, plate Ni,
Strip Thick PR in Acetone bath



boron membranes
use phosphorous doped substrate.

Wafer layout



= 14 devices (25x23) 100%
= 11 75%
= 7 50%
= 3 25% yeild on wafer

Use L-edit for Mask design.

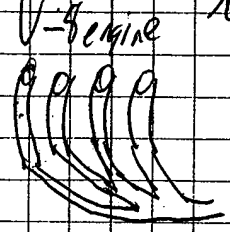
Need: KOH Mask -1
Bottom shell -2
Thick PR -3
Top shell -4

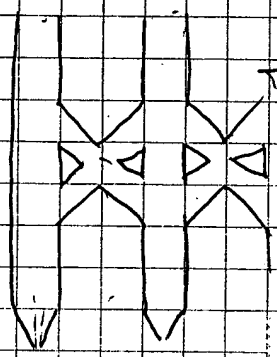
Cool idea!

90% Needles open per array
roughly equal velocity in each needle in equal pressure.
- From Tuned-port injection manifold use in later model Corvs.

So idea!

V-8 engine

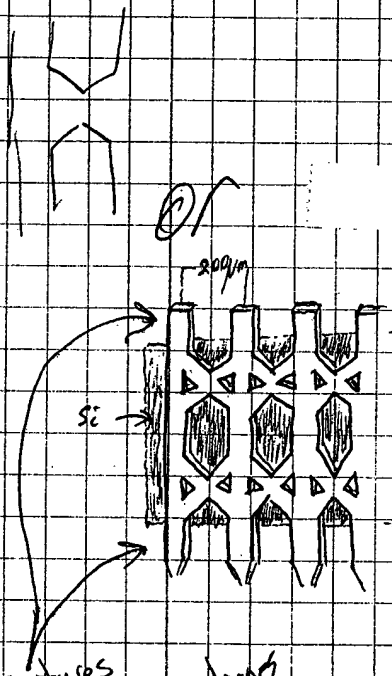




25 Needle array

Discussed with Frazier

Or

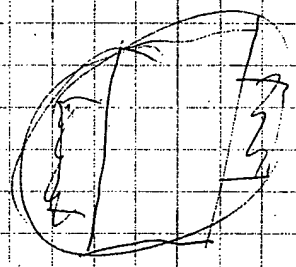


Mainly Nickel structure
Au or Ti inside channels for Bio compatability.

John D Bagley

Research use of Pd in place of Ni/Au structure.

Contact: Mr Marks @ Lucent Technologies, Bell Labs, 908-582-6408



John D Bagley

Pd coil literature Pd

Hardness: 430-500 KHN_{25}
purity: 99+ Pd
density: 11.7 g/cm^3
weight: 2.9 mg/cm^2 for 2.5 micron

John D Bagley

present palladium to group as possible replacement of e-plating Ni and Au.

John D Bagley

Think about mask dimensions

Mask Design

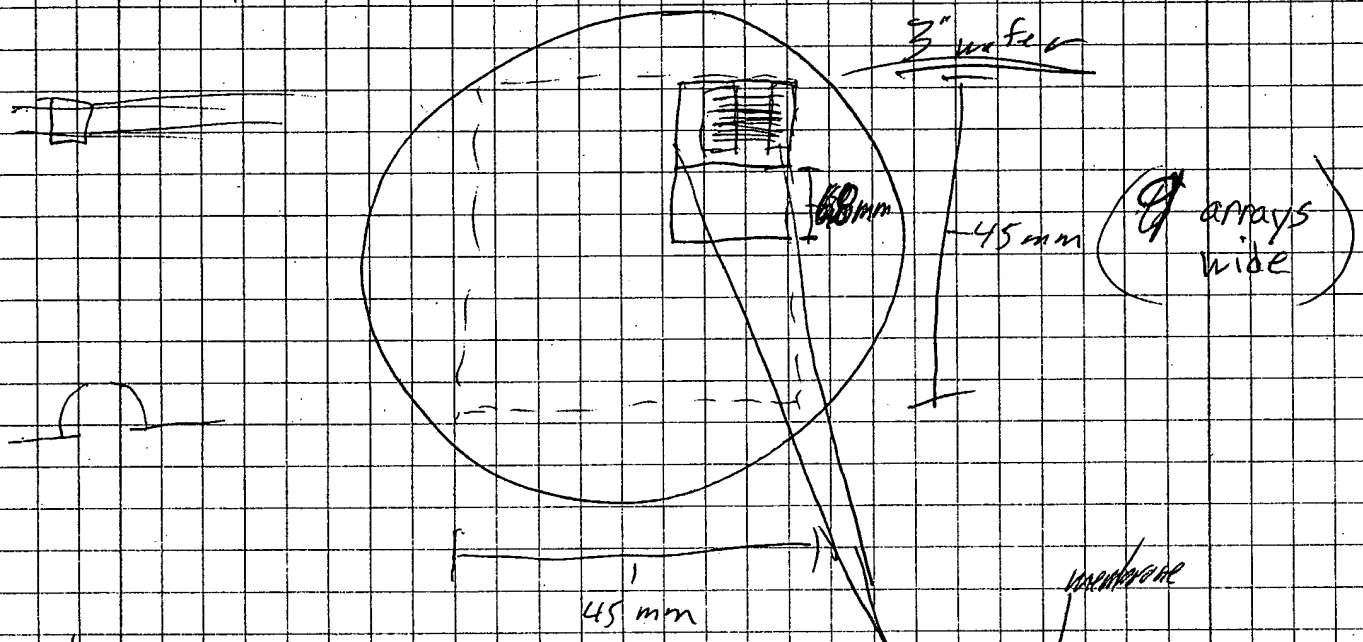
Mask Design — 3 masks total

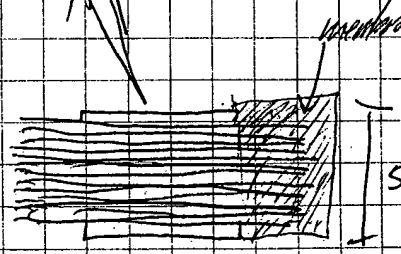
Bottom shell same as top shell

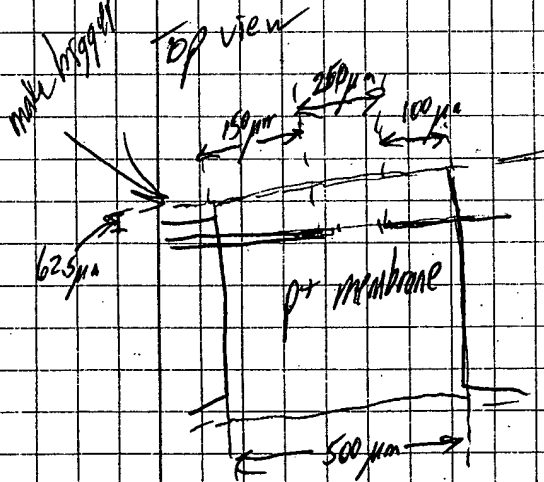
Mask #1 KOH Mask

- to define sacrificial membranes and alignment marks

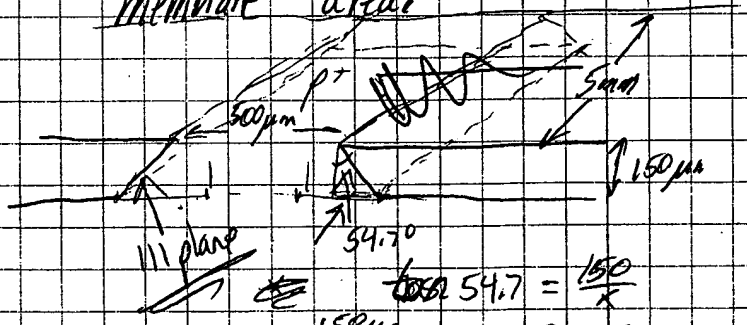
- using 45 mm² area on a 3 inch wafer







membrane area:

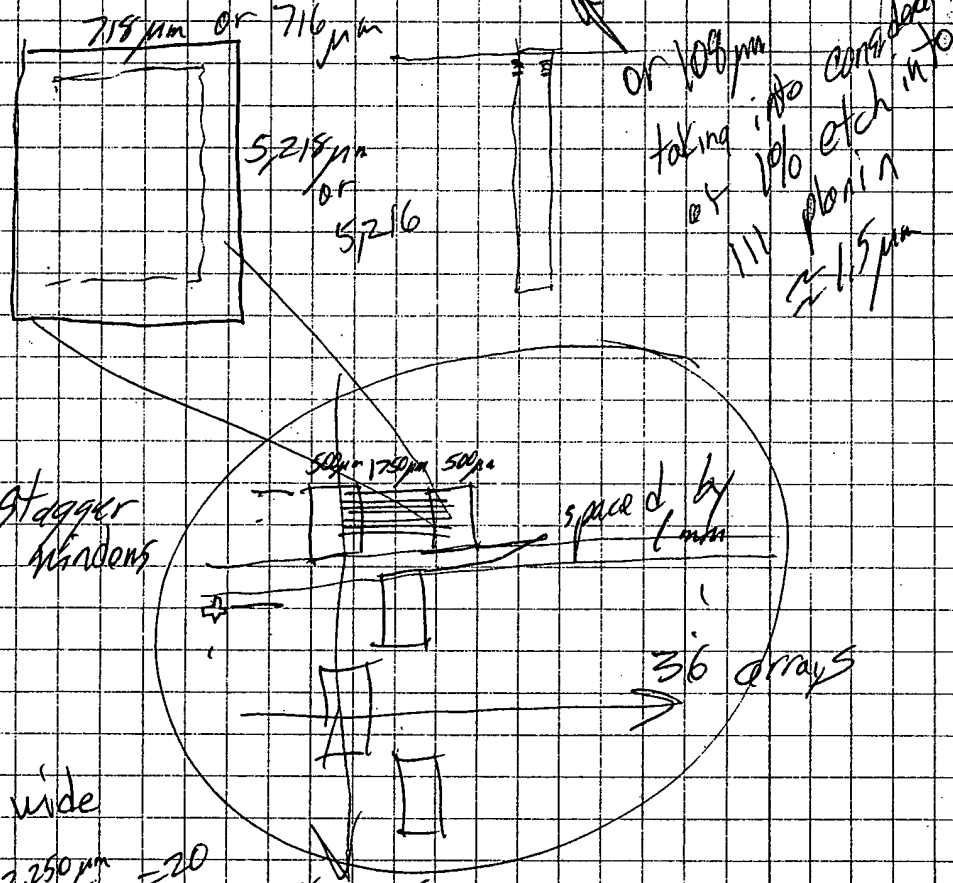


$\tan 54.7 = \frac{150}{x}$

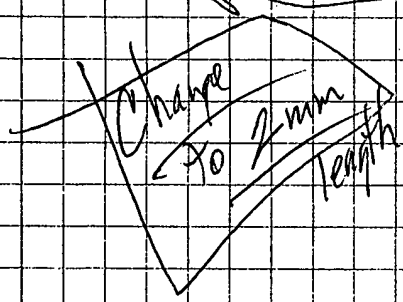
$x = \frac{150\mu m}{\tan 54.7} = 108.9\,\mu m$

Mask #1 continued:

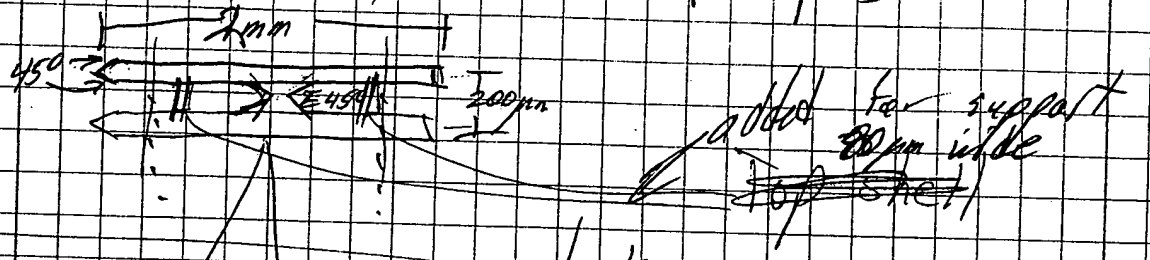
So window dimensions for mask are: width: 500μm + 2x = 500 + 2(109) = 718μm
and length: 5mm + 2x = 5mm + 2(109) = 5,218μm

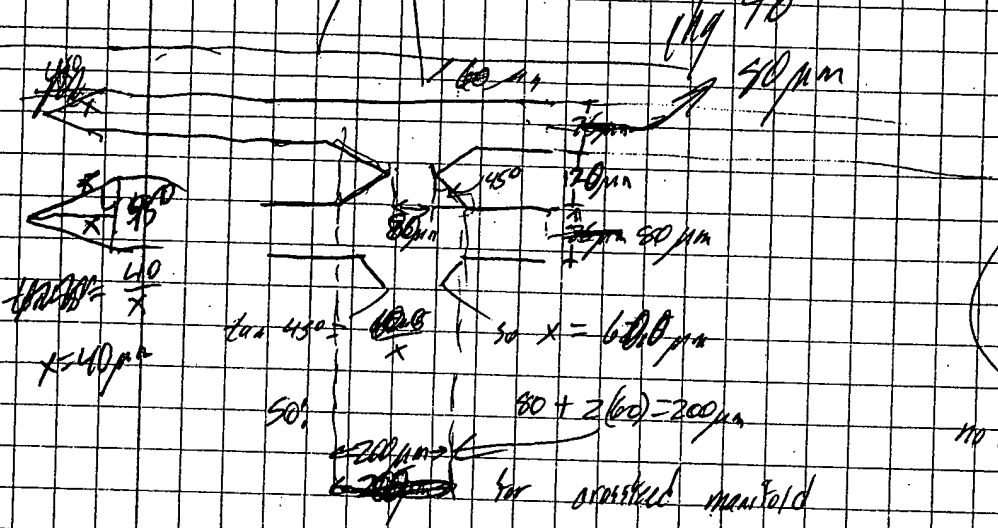


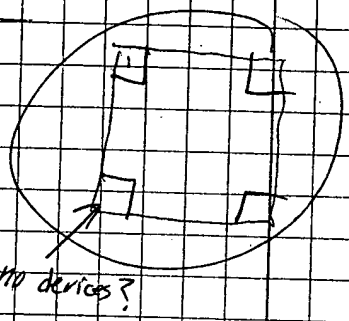
so 8 arrays wide
by ~~36~~ 19 long
19 long
2,250μm / 45000μm = 20



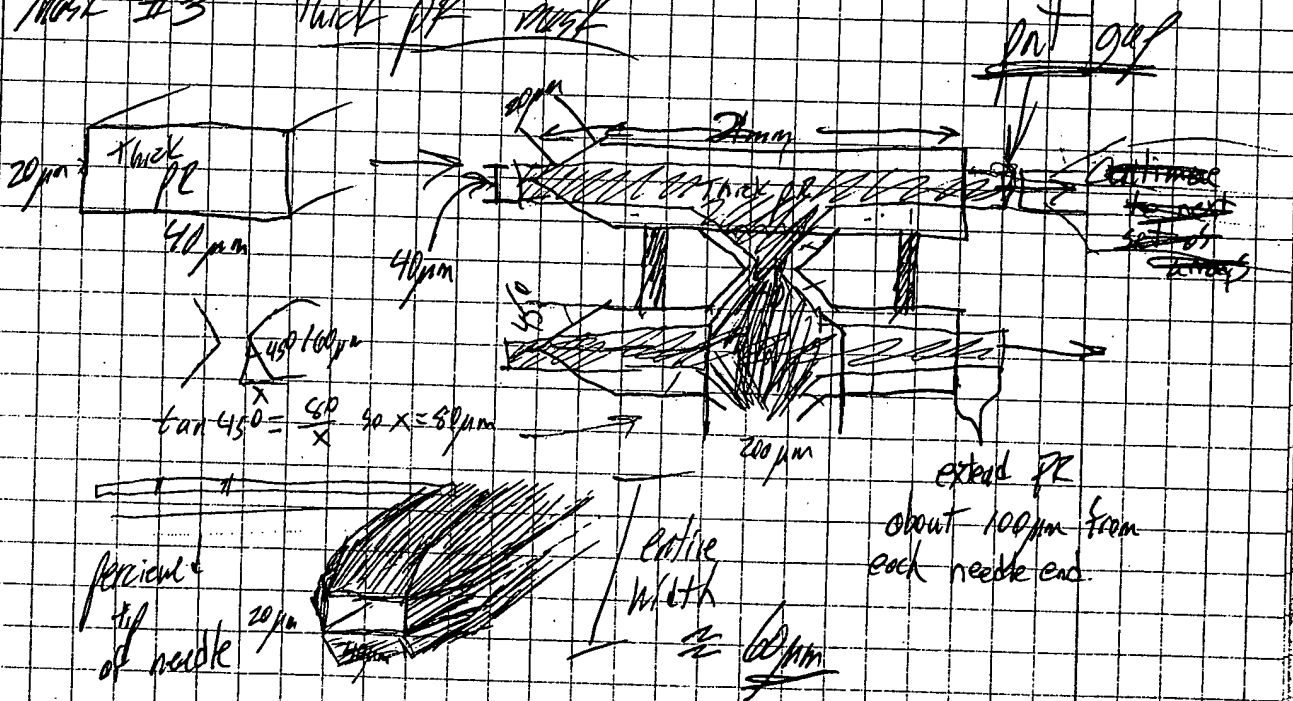
Mask #2 Bottom + top e-plating mask







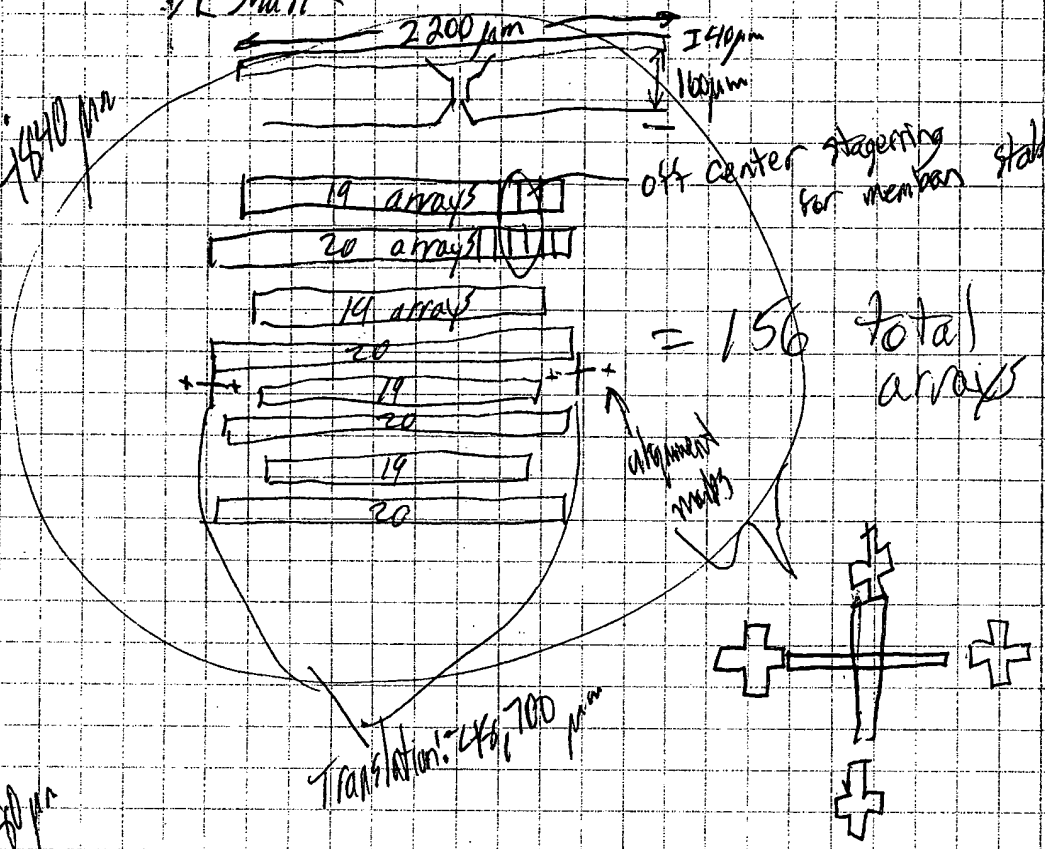
Mask #3 Thick PR mask

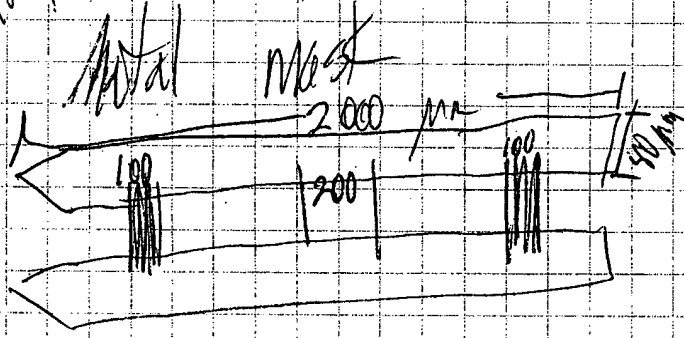


Qin & Engel

performing mask designs on L-edit

SR Mask

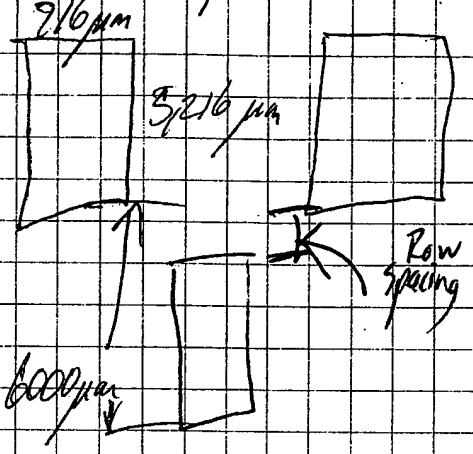




KOH mask

spacing: 1750 − 2(108) = 1534 μm

Dimensions:



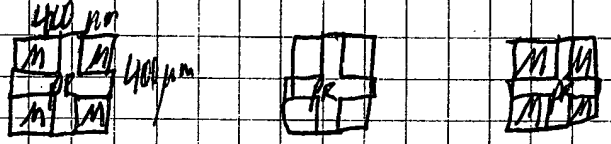
width: 3,216

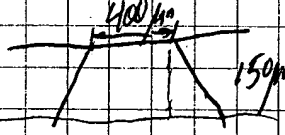
Translation: (for mask alignment)

x: −354

y: −189

Mask alignment marks:





x: −24,755
24,755
2455
1442

y:

$\tan 54.7 = \frac{150}{x}$ so $x = 108.98$ μm

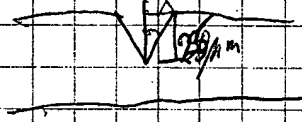
Dims are $400 + 2(108) = 616$ μm

so need 616 μm² alignment marks

Stripe marks:

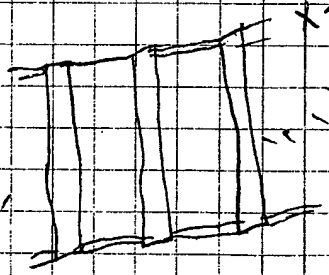
want to etch ~~25~~ 35 μm into sub strate

$\tan 54.7 = \frac{25}{x}$ so $x = 17.7$ μm



so make KOH lines 35 μm wide

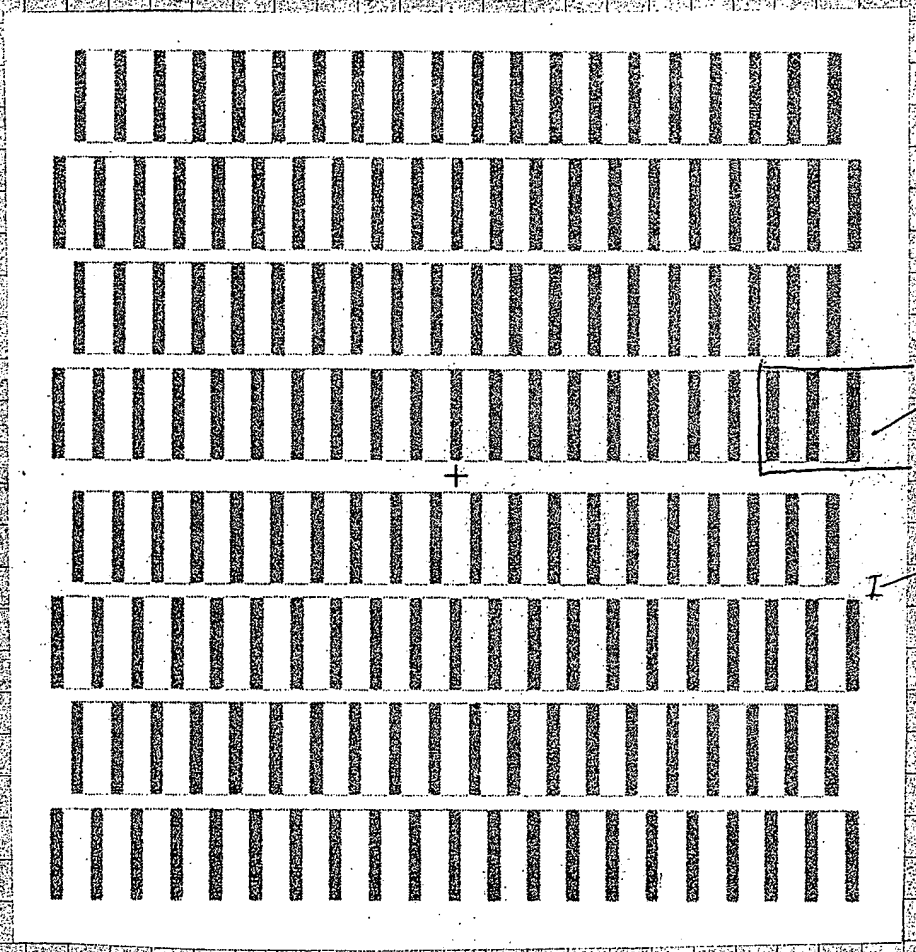
so:

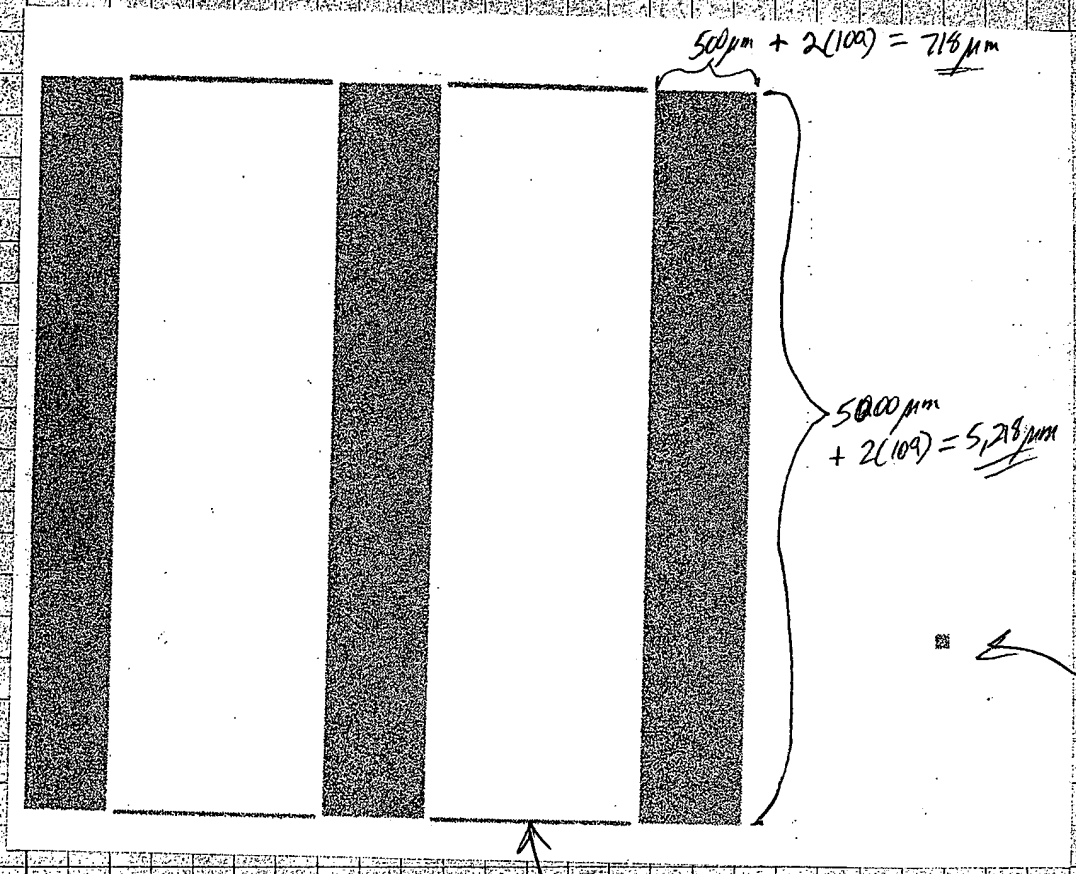


10

Completed Mask Design on L-edit

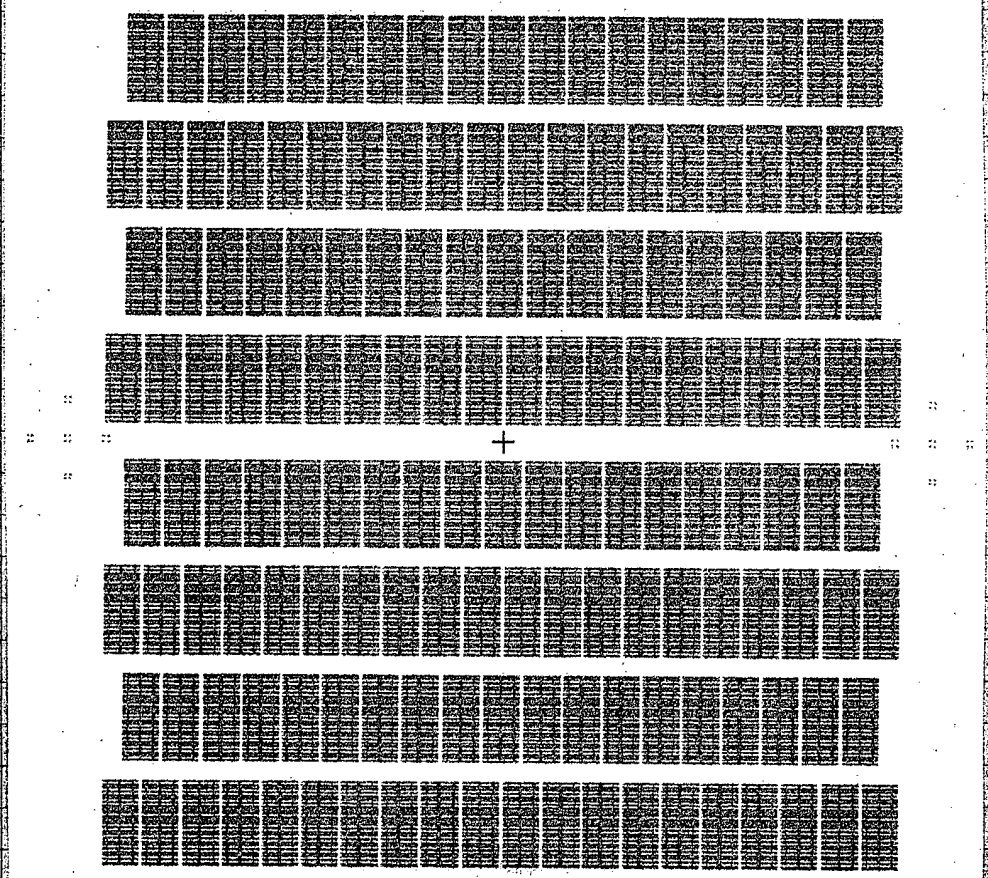
KOH Mask

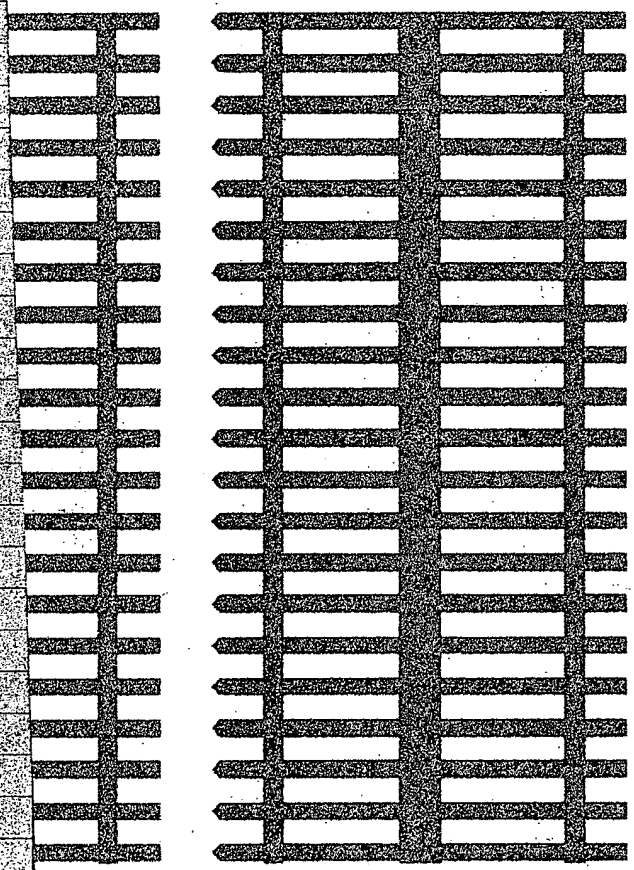



500μm + 2(100) = 718μm
5000μm
+ 2(109) = 5,218μm
Alignment Mark 100μm²
Scribe lines
35μm width

12

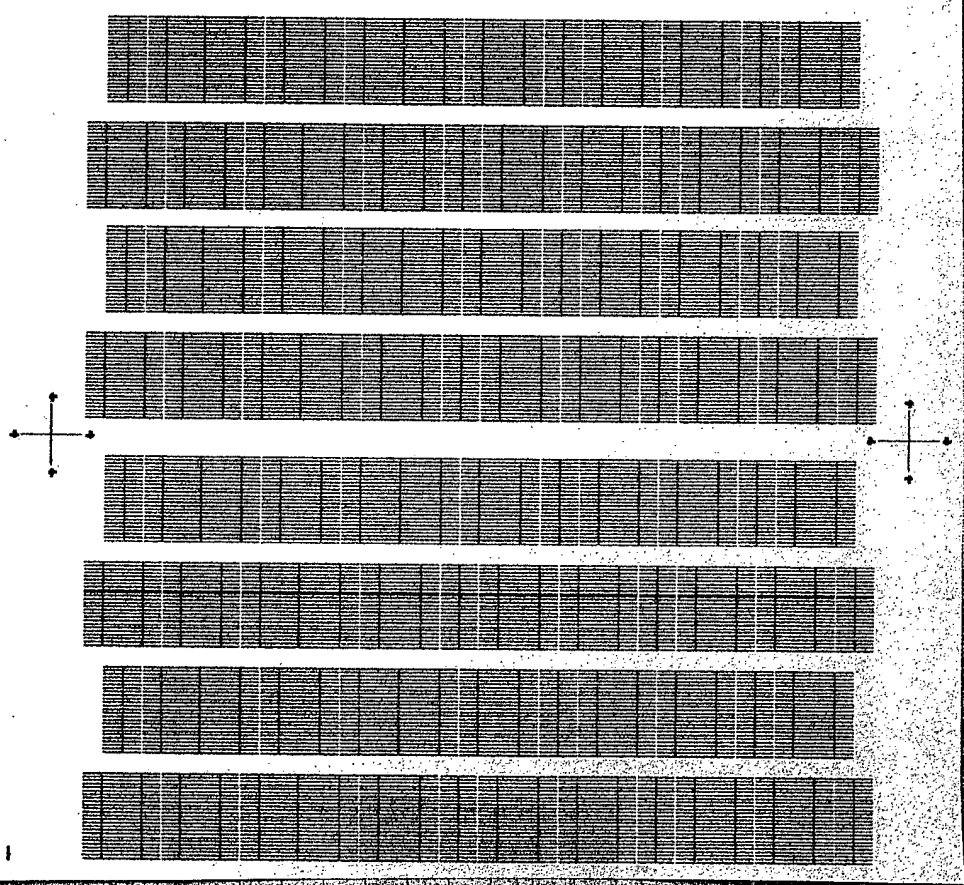
Metal Mask

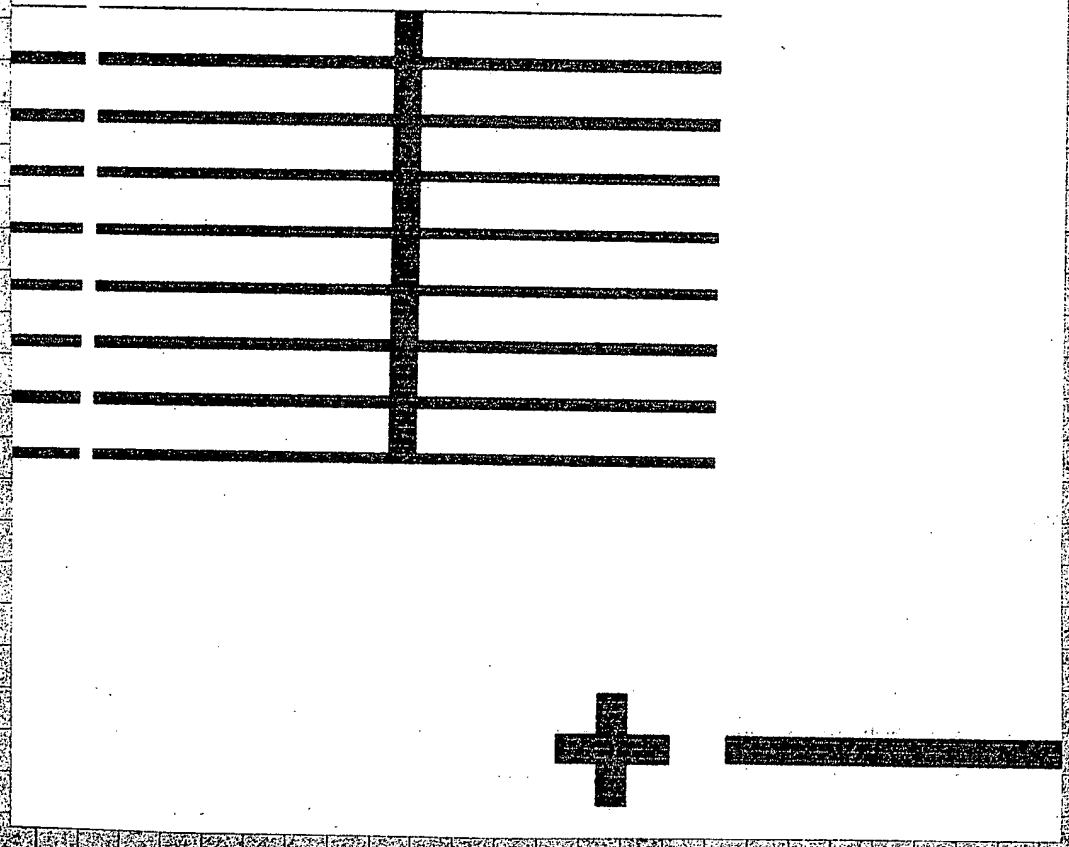




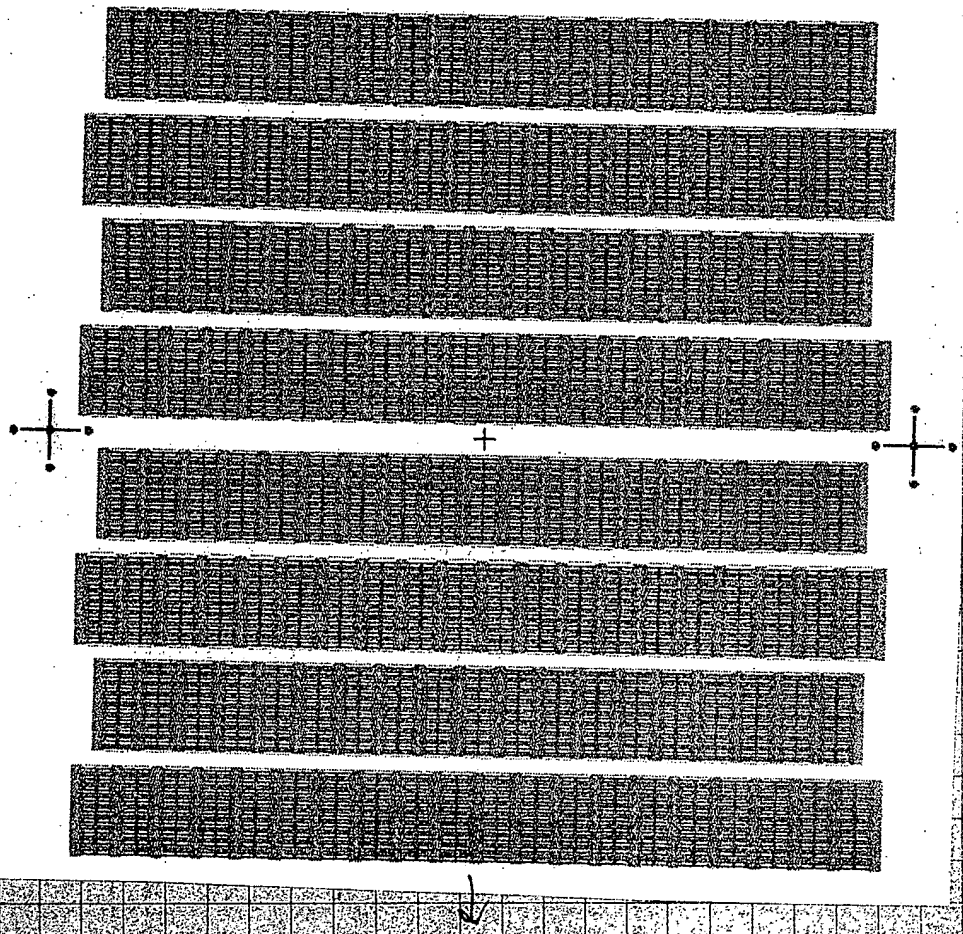
14

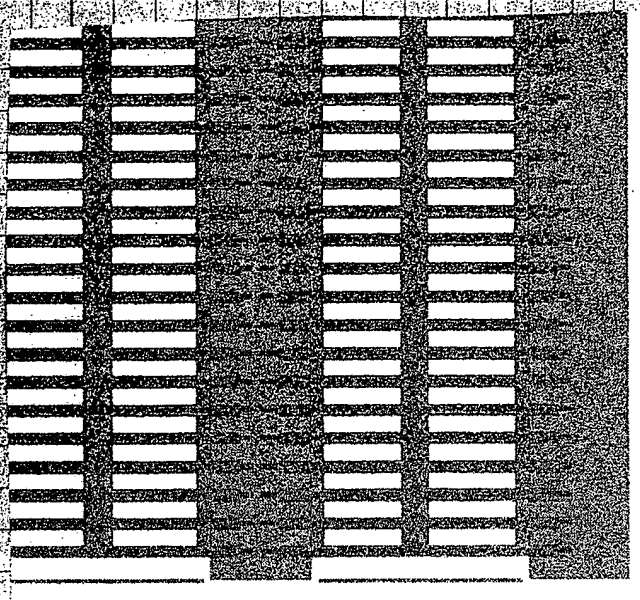
Photoresist Mask Etched

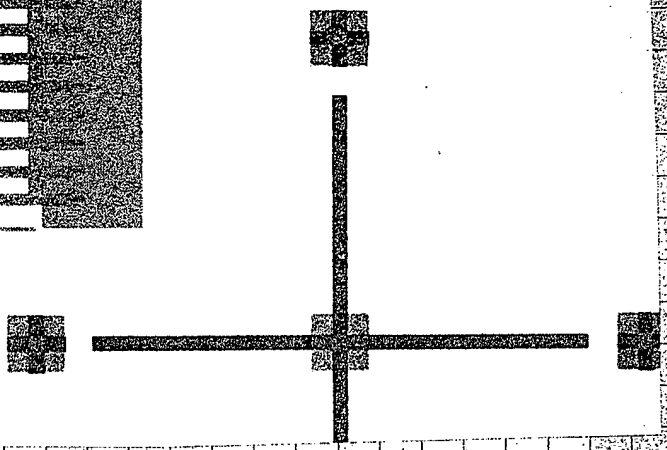


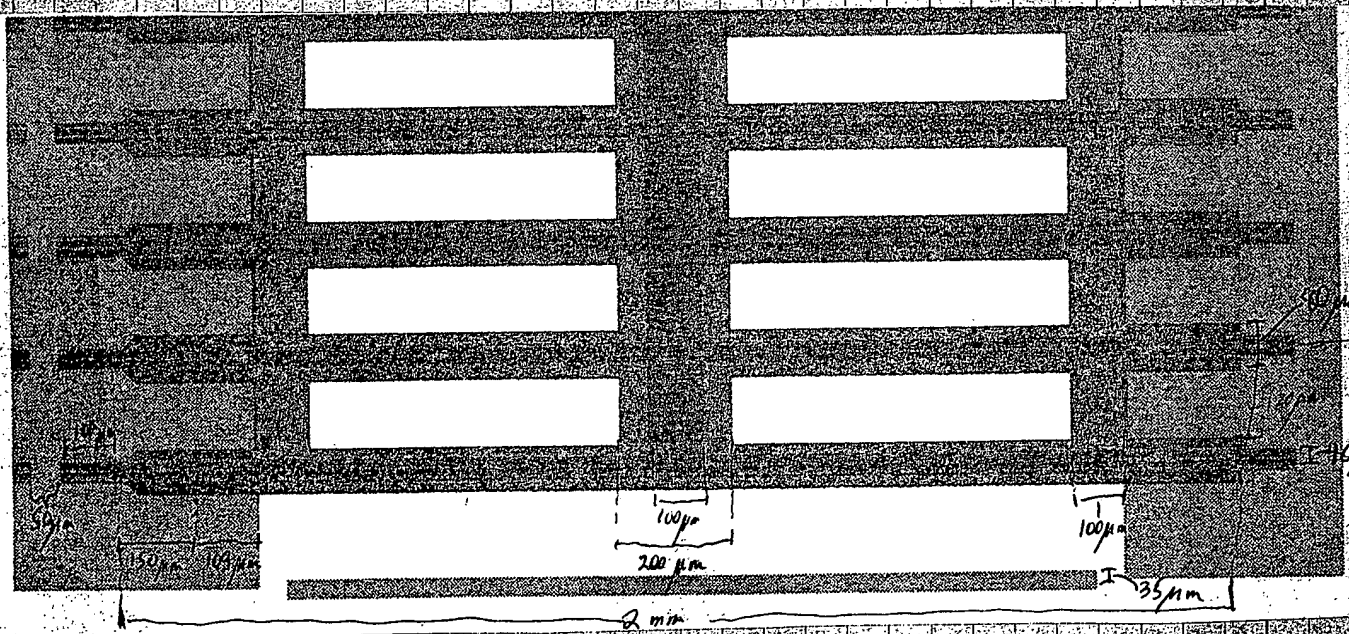


All Tree Masks

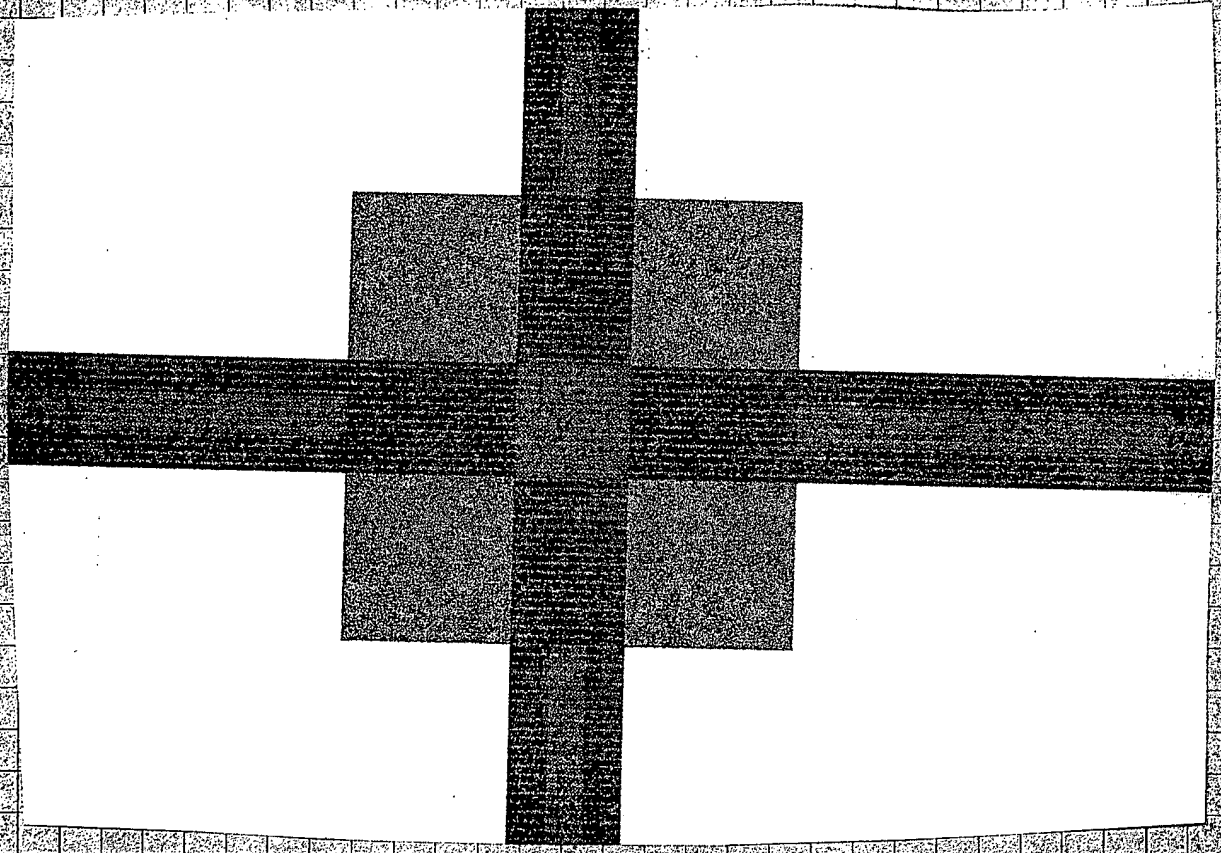







100μm
200 μm
100μm
35μm
2 mm

Alignment Marks (Centers)

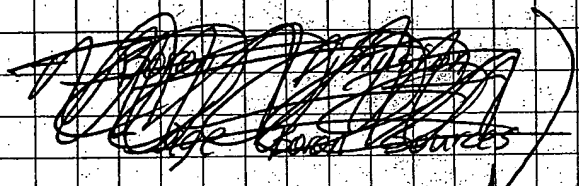


Masks in Fabrication

1- Wafer preparation (p+ Doping)

- Modified RCA clean

4 min piranha-etch 70 ml H_2SO_4 / 30 ml H_2O_2
- rinse DI 2 min
30 sec HF (5%)
- rinse DI 2 min



4 min Metal-etch 33ml HCl / 33ml H_2O_2 / 33ml H_2O
- rinse DI 2 min
30 sec HF (5%)
- rinse DI 5 min

* check wafers for hydrophobic character

- Boron Sources aging

800°C
80 steel O_2
95 steel N_2
5 hours

- Boron Diffusion (~ 3μm)

1175°C
11 glass O_2
131 steel N_2
5 hours

3- Prep. for Si_3N_4 deposition

- HF dip (6%) 3 min
- DI Rinse 5 min

+ Si_3N_4 deposition using PECVD

Substrate temp: 300°C

N_2 flow: 15 sccm

NH_3 : 55 sccm

SiH_4 : 23 sccm

LF power: 40 w - ~~3~~ sec pulsed

HF pwr: 4 w - 4 sec pulsed

pressure: 300 m Torr

Time: 45 min each side of wafer

4- KOH etching (anisotropic)

- pattern:

PR 1813 ~~1500~~ rpm 30 sec

100°C 40 sec

expose: 15 sec

develop: AZ 352 ≈ 30 sec

- RIE Nitride etching

CF_4 flow 40% (40 sccm)

power 50% (150 w)

pressure 300 mTorr

Time ≈ 8 min

- water clean

Acetone 15 sec
Methanol 15 sec
piranha-etch 2 min 70% Sulphuric 30% H2O2
DI Rinse

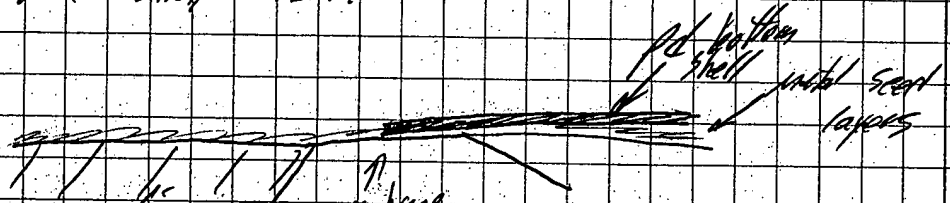
- KOH Bath

~~300~~ g KOH to 1200 ml H_2O
Temp: 60°C
Time: ≈ 7.5 hours
Note: slow agitation of bath mixture w/ plastic mesh to break-up flakes

- DI Rinse 5 minutes
N_2 blow dry (low pressure)

4 ~~Mon~~ 97

5- Bottom shell formation



- Metal seed layers (Sputter)

Power: ~~250~~ W
Argon: 60%
Vacuum: 1×10^{-6}
Time:
Ti → 4 min ≈ 600 Å
Cu → 2 min ≈ 1500 Å
Cr → 3 min ≈ 1000 Å

→ 90 to 95 for process later

22

Thick PR process development

1st attempt: (on Gold covered wafers)

PR 4620

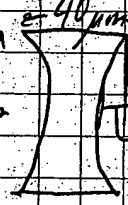
300 rpm 15 sec
900 rpm 20 sec

Cure: 110°C 2 min

Expose: one bar 20 sec
" " 30 sec

develop: 40 ml 400K : 100 ml H2O
~ 5-8 min

Results:



~~5μm~~ each side

30 sec exposure looks better; (possible reflection problem)

2nd attempt: Apply Anti-Reflective Coating

AR-L

1600 rpm 20 sec
~~2000~~ rpm 60 sec

Bake: ~~130~~°C 20 s
145°C 1 min

PR 4620

300 rpm 15 sec
950 rpm 30 sec

Cure 110°C 2 min

Expose: 40 sec, 30 sec, 20, 10

develop: 400K

Bad Results

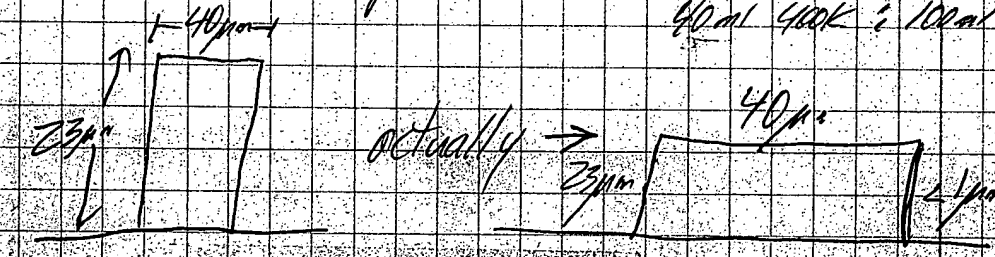
3rd attempt. used
50 s 40 s 30 s 20 s
110 RPC
40 s looks best

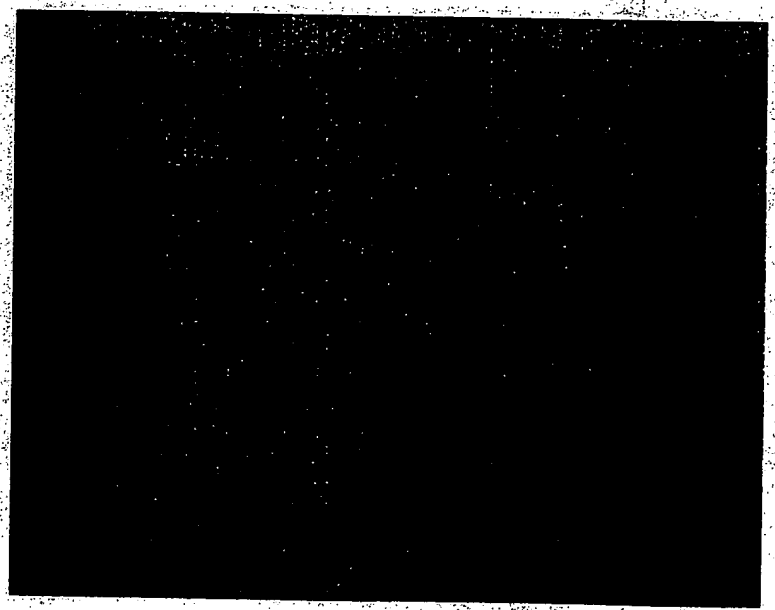
4th attempt 40 sec looks perfect.

procedure:

PR 4620 300 rpm 15 sec
950 rpm 20 sec

Cure 110°C 2 min
Expose 40 sec
Develop 400K ~~[illegible]~~ 2.5:1
40 ml 400K : 100 ml H_2O





Sputter on thick PR

Attempts: 1 - 10 min @ 100W RF ≅ 1200 Å

Failed - left bubbles in PR

2 - 5 min @ 100W RF ≅ 600 Å

Failed -

3 - 2.5 min @ 100W RF ≅ 300 Å

GOOD!

Need ≅ 1000 Å

So:

Use this ☆ 4 - 2.5 min @ 100W RF
then 15 min cool
2.5 " " " "
" 15 min cool
the 2.5 min " "

yields ≅ 1000 Å Good appearance

Thick PR over encapsulated PR

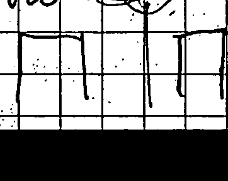
4620 spread 300 rpm 20 sec → Good
1000 rpm 20 sec

bake 37°C (oven) 25 min → Good

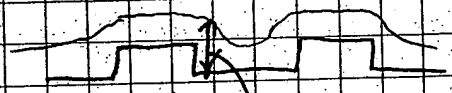
Expose 40 sec ← Bad

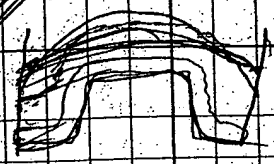
Develop 400K ~~3:1~~ 2.75:1 30 ml 400K, 100 ml H_2O

Did not work Next try 50 s + 60 s



Suspected Underexposure





this region is thicker than 23μm
make 27-35 μm so increase
exposure next time.
—problems with water sticking to mask
need to cure longer
—Trial @ 35 min cure time
results: Good cure
exposed for 55 sec NOT Good enough
need to increase

Take 2 Exp

All Dummy wafers trashed!
Have 2 wafers w/ Ti/Cu/Cr
Pattern for bottom shell

—PR 4620 300 rpm 15 sec
1000 rpm 20 sec
Bake 110°C 2:15
expose 45 sec
Develop 35ml 400K / 100ml H2O
results: Good side wall appearance!

—E-plating of bottom shell
Cr-etch 75% HCl / 25% H2O
Pd plating 30°C questionable bath?
Current Density: 5mA/cm² Area = 30.37 cm² Total

Pd plating cont.

5 ma/cm² × 8.04 cm² = 40.2 mA current setting

plating Time: 1 hr ≈ 12 μm

Results: Adhesion problem

possible ~~tension~~ ~~compressive~~ stresses

tensile (peel-up)

at 3.73 ma/cm² × 8.04 cm² = 30 mA current setting

plating Time: 45 min

~~Bad~~ Adhesion

Bottom shell w/ 1 hour nickel and 10 min Pd

w/ membranes; bad 1000 rpm, 30 sec neg PR, 3 min @ 110°C

pattern Thick PR; exposure at ~~50 sec~~ was better

Thick PR on bottom shell;

PR 4620 300 rpm 15 sec

1000 rpm 20 sec

bake 110°C 2:15 min

expose 55 sec (on membrane wafer)

develop: 35 ml 400K/100 ml H2O

Note: Exposed for 45 + 50 sec on surface wafers.

50 looks a little better

Sputter:

100 w RF Au for 2.5 min

Cool 15 min

100 w RF Au for 2.5 min

Cool 15 min

100 w RF Au for 2.5 min

Note: 50 sec exposure looks best

Top Shell Microvia'd

JK 4620 300 rpm 20 sec
1000 rpm 20 sec

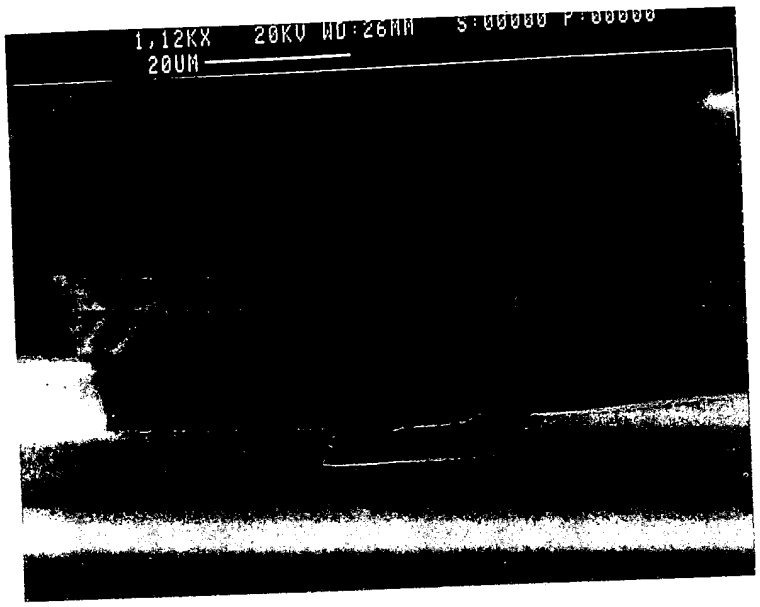
Bake 37C (oven) 35 min
Expose: 60 sec
Develop 35 ml 400K / 100 ml H_2O

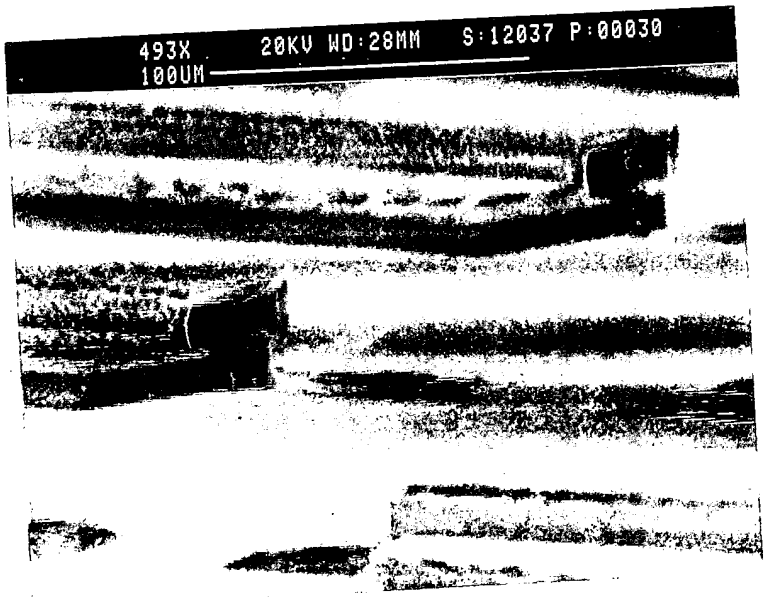
Stuck to mask
Increase cure time

Top shell Cure time @ 60 min @ 37C
100%
Expose: 60 sec
Develop 400K
Good!

Top shell electroless
10 min Pd @ 5 mA/cm²
1 hour Nickel @ 10 mA/cm²

SEM of Surface work





Notes: Awaiting better wafers from Silicon Quest. Expect arrival on 14 Dec.

~~14 Dec~~

If wafers in bath are trashed!
Sizing lifted completely off the unpolished side.

14 Dec 97 ~~14 Dec~~

Took two wafers and did 1.15 hrs of Sizing on the unpolished side and 45 mins on polished side.

20 Dec
Both @ 60C start time 1am.

300g KOH / 1200 ml H_2O

Results: after only 3½ hours, the KOH was etching the entire surface due to roughness of the unpolished side.

~~Alternate~~ Alternate Technique: Using one of the existing wafers w/ completed surface with lift-off procedure

etch Chromium: 50% HCl @ 2 min
etch Copper: Ammonium Hydroxide saturated with Cupric Sulphate. @ 1.5 hours

Backside of wafers too rough.
Try SiO2 as mask for KOH
Need to characterize a 3" furnace.

John D Light

Furnace failed several attempts. Furnace sound to be malfunctioning.
Parts on order

John D Light

3" Furnace still inoperative. Attempting to fire up new 4" Furnaces - awaiting electricians, starting ANSYS finite element modelling of the needles.

John D Light

Met w/ Frazier.. decided current wafers will not work. Double-sided polished flexible wafers on order. Also will use lift off technique to see if assembly is possible.

Lift-off procedure for Wafer fabrication -

started w/ 6, 10-12 mil thick, new wafers
performed standard water clean.

Deposit Metal seed layer system (Sputtered)

Power:	250 W
Argon:	60%
Vacuum:	1.2×10^{-6}
Pressure:	4.77
Flow:	55.7

Times + Metals

Ti → 4 min ≈ 600 Å
Cu → 3 min ≈ 2250 Å
Cr → 3 min ≈ 1000 Å

Pattern Bottom Shell

PR 4620

300 rpm	15 sec
1000 rpm	20 sec
Bake 100C	2:15
expose:	45 sec
develop	1:4 400K / 100 ml H_2O

Bottom shell e- forming

etch Cr in 25% HCl / 25% H_2O

Pd bath: pH: 8.1
Current Density: 5 mA/cm²
Current Setting: 5 mA/cm² × 8.04 cm² = 40.2 mA
Plate time: 1 hr yields: Adhesion problem

Adhesion Problem investigation

- found pin-holes in Cr layer, etch didn't remove all Cr before plating. possible more layer of Si3N4 on the Chromium.

prepared new batch of wafers

1 - Clean
- Si3N4 both sides
- Ti, Cu, Cr
- patterned 2 wafers w/ bottom shell

wafer #1
plate @ 40.2 mA = 5 mA/cm²
Time: 1 hr
Thickness: ≈ 3 μm
Results: GOOD Adhesion

Wafer #2:

Plate Pd @ 80 mA = 10 mg/cm²

Thk ≈ 10 μm

Good adhesion but very rough!

Patterned 2 more wafers w/ bottom shell

Wafer #1:

Current: 40.2 mA

Time: 3 hours

Thickness: ≈ 17 μm

Results: poor adhesion (possible PR problem)

Same as #2 wafer

Must solve Pd adhesion problem

Bottom shell Sprayed w/ nickel

1 hr @ 120 mA (15 mA/cm²) yielded ≈ 18 μm thick

PR on bottom shell

300 rpm 15 sec, 1000 rpm 20 sec, bake 110°C 2½ min

expose 50 sec, develop 450 ml 400K/100 ml H₂O

Sputter Au

100 w RF 2.5 min
Cool 15 min
100 w RF 2.5 min
Cool 15 min
100 w RF 2.5 min

~~Top shell microMold~~

Prepared two more wafers as above.

Top shell microMold

PR 4620 300 rpm 15 sec
1000 rpm 20 sec

Cure in oven @ 37°C for 60 min

~~Expose~~ 60 sec

Develop 100ml H2O / 40ml developer

Top shell electroforming
1 hr nickel @ 100 mA (15 mA/cm²)

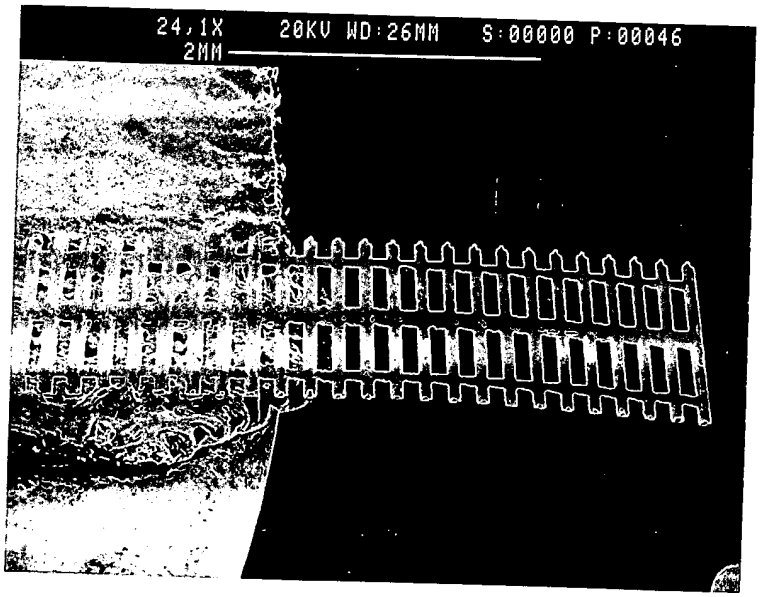
Results: Good!

Glass Slide

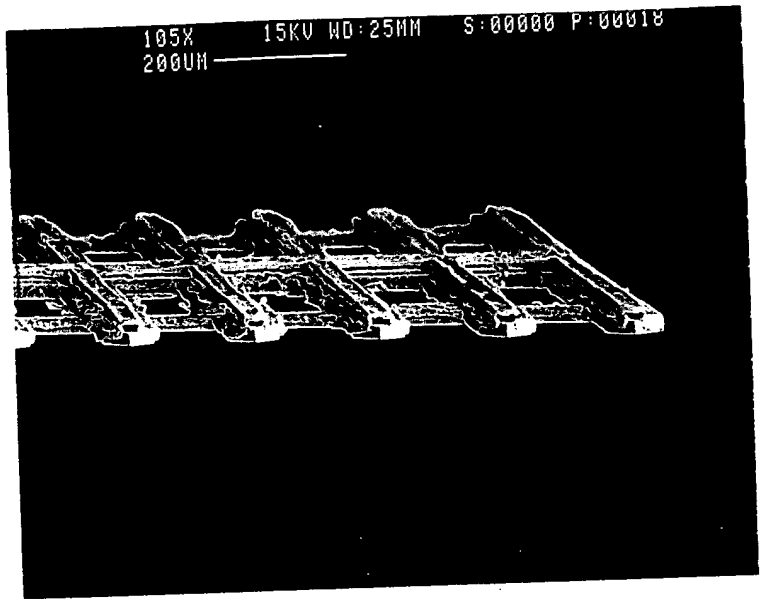
Lift-off procedure (Dry between each step)

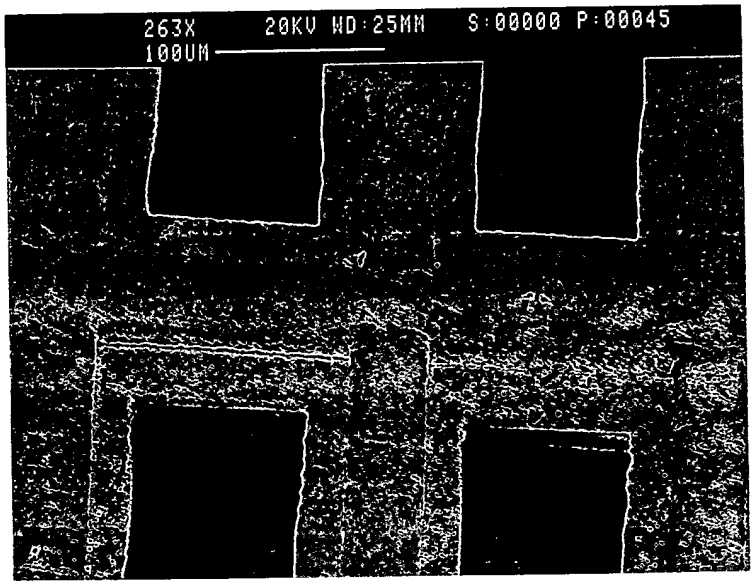
1- 30 sec "Gold etch"
2- 5 min Acetone
3- 2 min 2-propanol
4- Cr etch in 75% HCl ≅ 2 min
5- Copper etch to lift-off ≅ 1.5 hours
(Cupric sulphate saturated in Ammonium Hydroxide)

preparation for SEM.

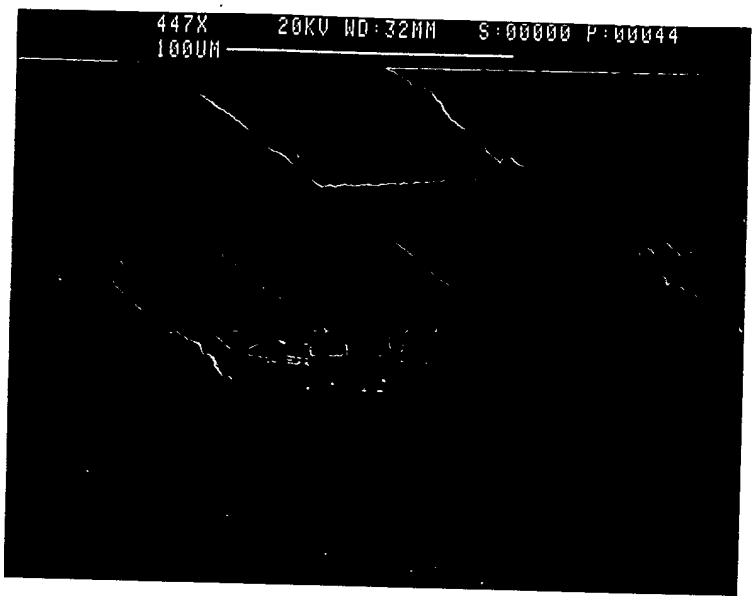


Array of 25 needles



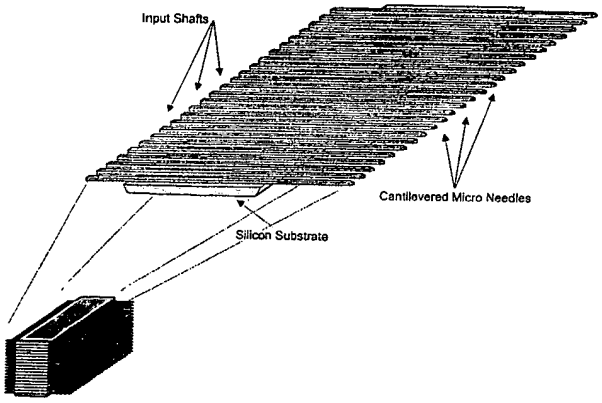


Needle Coating Channels



$\approx 20 \times 30\ \mu m^2$ ish

Consider Methods for assembly of 2D devices into a 3D device.

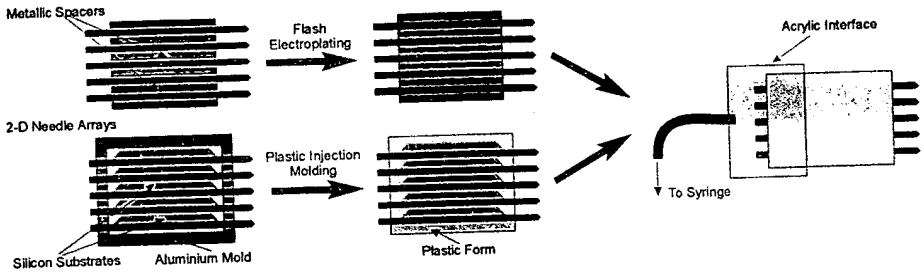


Possible methods for assembly into a 3-D device:

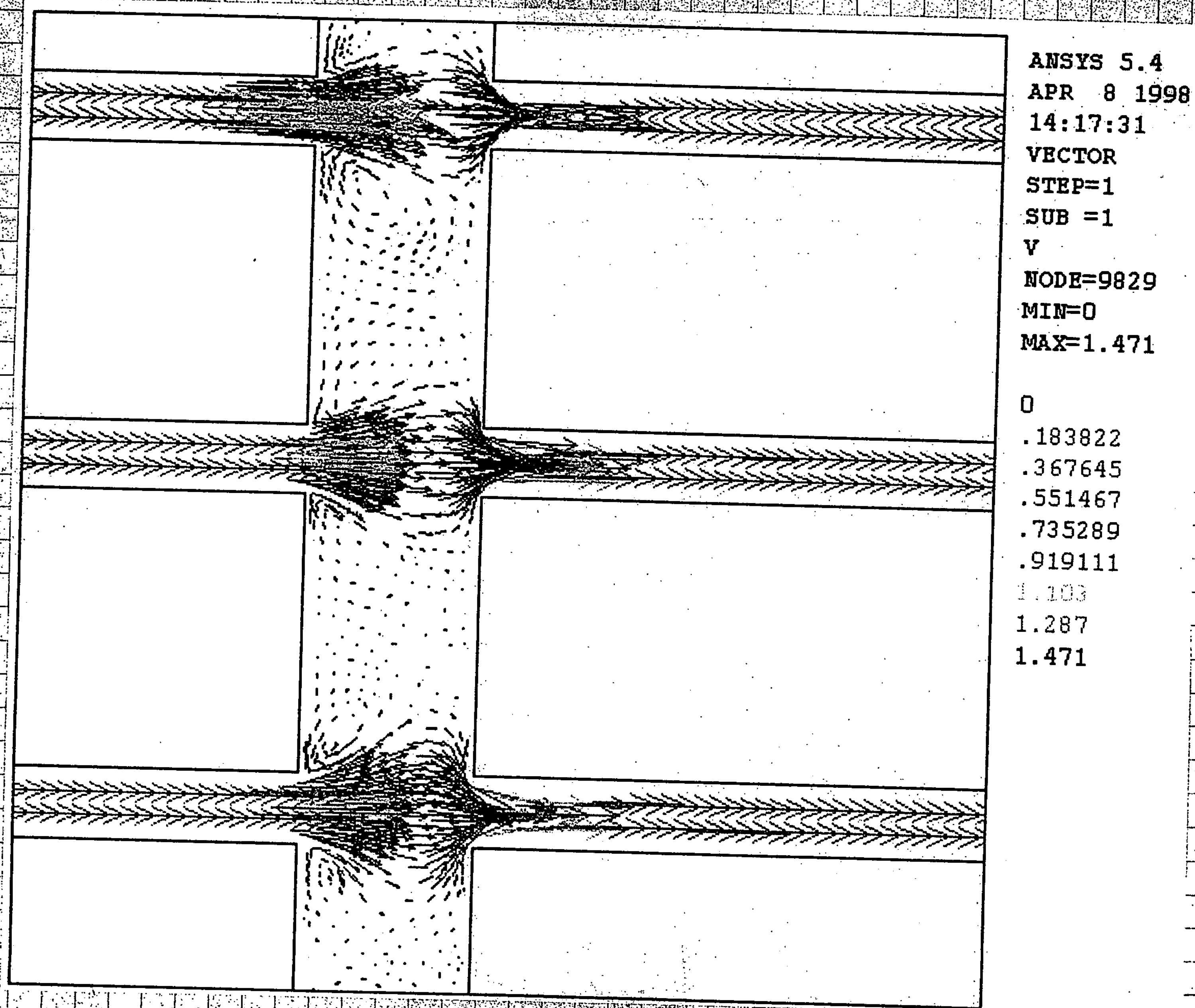
1- Use 150μm thick, double-sided tape.

2- Flash-plating w/ released devices and metallic spacers.

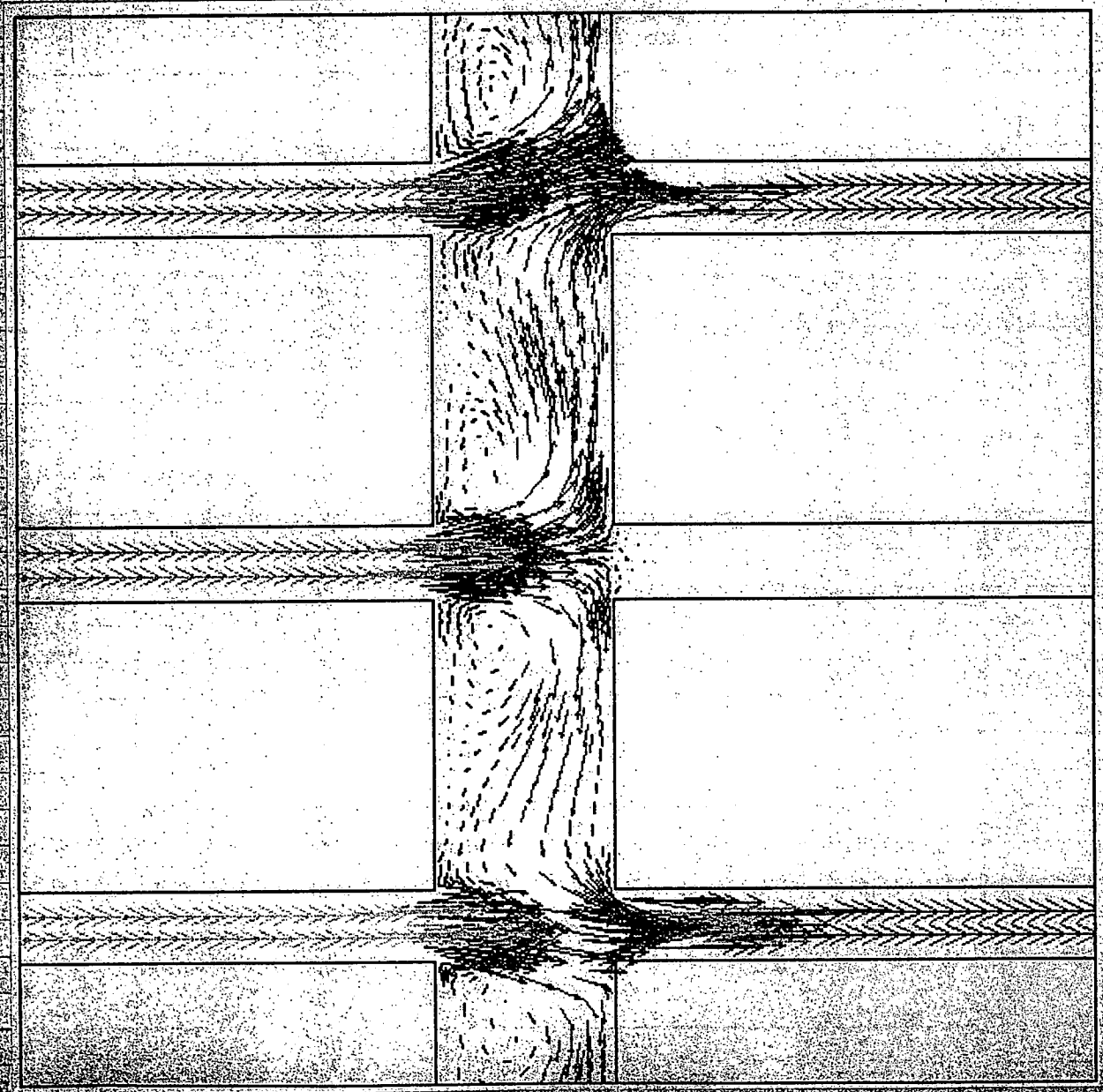
3- Plastic injection molding w/ aluminum mold.

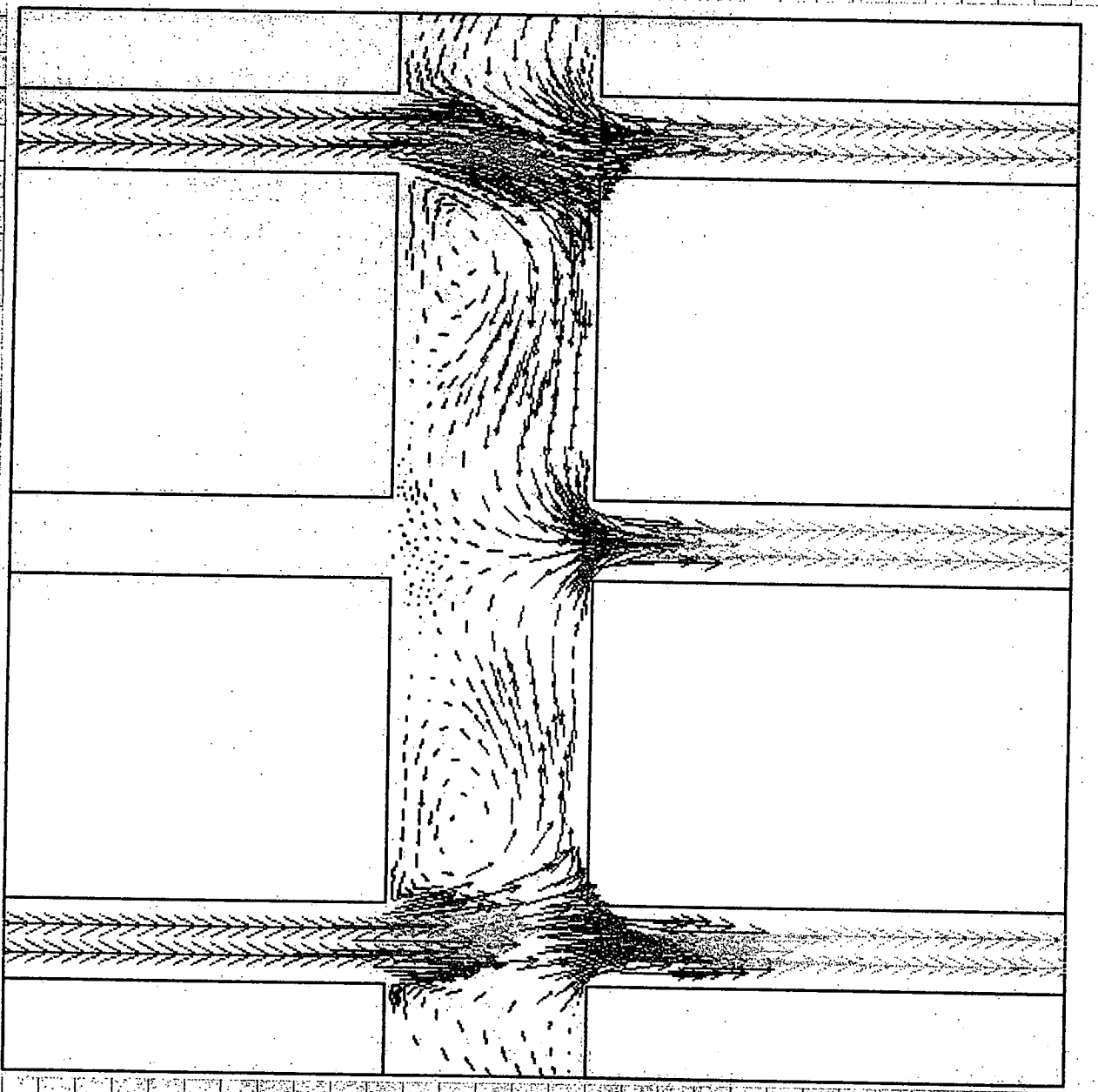


ANSYS Modeling to determine fluid flow through fluid coupling channels



no deg


ANSYS 5.4
APR 8 1998
13:39:02
VECTOR
STEP=1
SUB =1
V
NODE=8660
MIN=0
MAX=1.661
0
.207641
.415283
.622924
.830565
1.038
1.246
1.453
1.661


ANSYS 5.4
APR 8 1998
14:43:45
VECTOR
STEP=1
SUB =1
V
NODE=3675
MIN=0
MAX=1.459
0
.182355
.364711
.547066
.729421
.911776
1.094
1.276
1.459

John D. Lyl

- assembly method attempted manually under microscope using double-sided tape 150 μm thick.

Results: Fair to poor!

25 needles arrays stacked

Still pursueing other methods.

* Interface for single 2-D array is ~~(FAB)~~ under manufacture.

List of items to be accomplished:

1- Bond 2-D array to single interface and test w/ syringe.

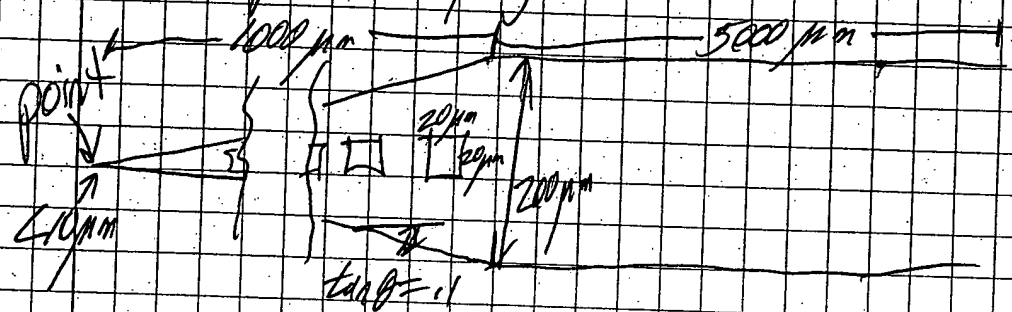
2- Fabricate grid/mesh for assembly of 2-D arrays into a 3-D device.

3- Bond 3-D device to interface and test w/ syringe

New Needle Design

(previous is considered old design)

— Tapered Syringe Needle



Usage 919-990-2228

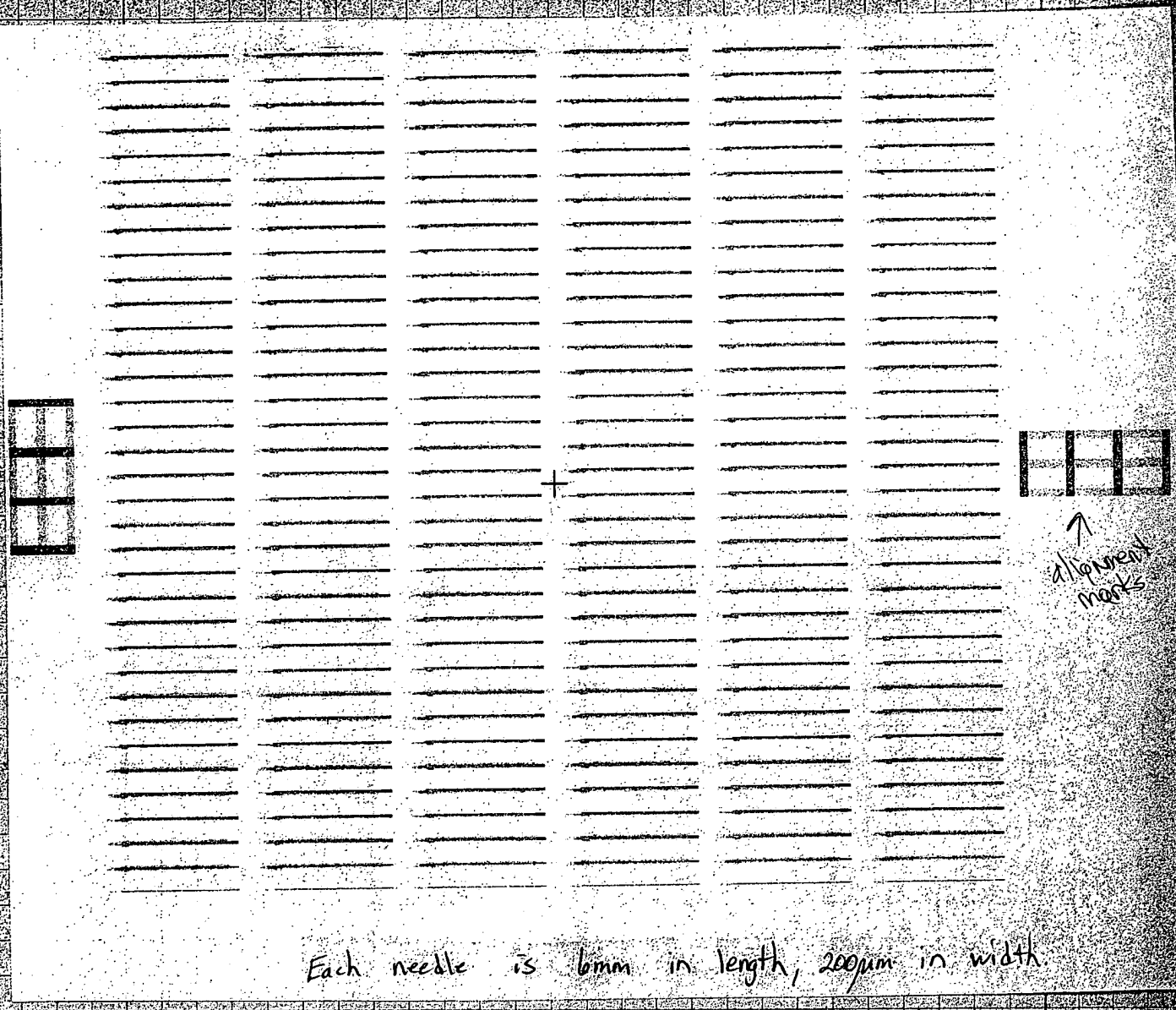
Notes:

1) Square crossection

2) along tapered section: place 20×20 μm² opening at d = 250, 300, 350, ... 1000 μm. on two to four sides

14 9 July '98

Completed Mask Design



Each needle is 6mm in length, 200μm in width.

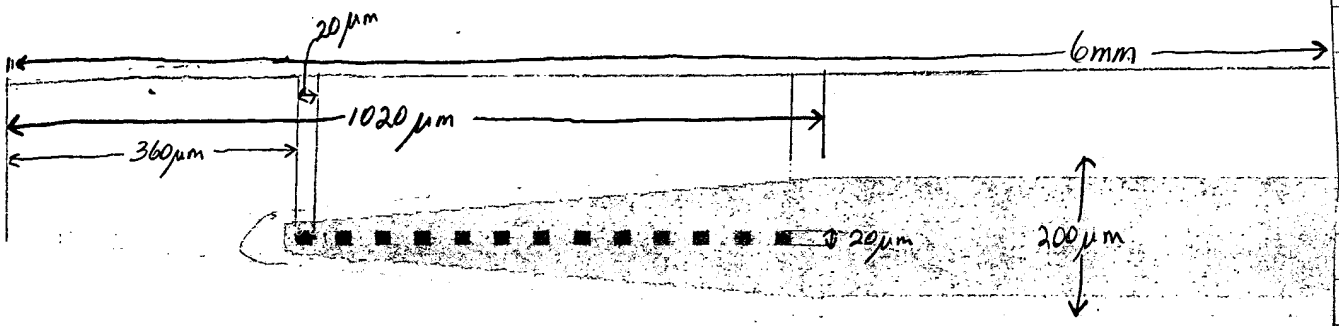
Masks are complete!

6 columns of 35 each needles

Bottom shell area = ~~[illegible]~~ ~.55cm x .02cm x 35 x 6

≈ 2.31 cm²

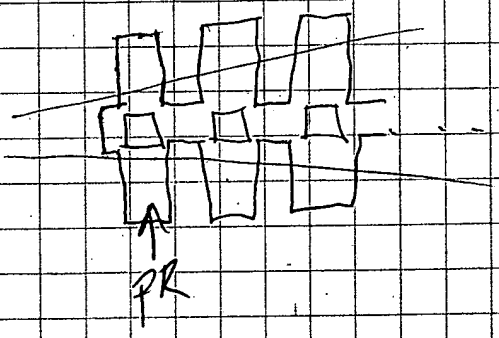
+ alignment [illegible]



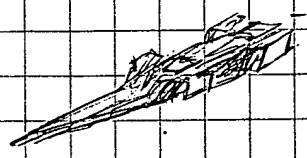
Note: approximately 20μm is expected to be lost on the needle tip length due to 'intentional' processing techniques.

This design allows for openings on TWO of the four sides.

For openings on four sides. Simply modify PR mask to:



should get:



July 98

Fabrication for lift-off of new design:

Wafer prep
5 wafer

Clean: 4 min piranha-etch 70ml H_2SO_4/30ml H_2O_2
2 min DI rinse
30 sec HF (5%)
2 min DI rinse
4 min Metal-etch 33ml HCl/33ml H_2O_2/33ml H_2O
2 min DI rinse
30 sec HF (5%)
2 min DI rinse
ensure hydrophobic character
SO3/4 on Both sides of wafer

Bottom shell formation

Sputtered seed layers
Power: 250W
Argon: 60%
Vacuum: 1.2×10^{-6}
Pressure: 4.7
Flow: 55.7
Metals: Ti → 4 min → 600Å
Cu → 3 min → 2250Å
Cr → 3 min → 1000Å

Pattern Bottom Shell
PR 4620 3000 rpm 15 sec
1000 rpm 20 sec
Bake 110°C for 2:15
expose: 45 sec
Develop 40 ml 400K/100 ml H_2O results: lost some parts

10 July 98

E-plate Bottom shell

etch Cr: 75% HCl

Ni: 15 mA/cm² × ~2.5 cm² ≈ 37 mA

Time: 1 hr ≈ 17–18 min

Pd: 5 mA/cm² × 2.5 cm² ≈ 12.5 mA

Time: 10 min

Strip PR in Acetone w/ alcohol rinse

Thick PR on Bottom Shell

PC 2620 300 rpm 15 sec
1000 rpm 20 sec
bake: 110°C 2:15
expose: 50 sec
Develop: 40 ml 400K / 100 ml H_2O

Sputter Au

100 W DC for 3 min
substrate on top of Soft
for heat dissipation

13 July 98 — John O'Brien

Top shell fabrication

PR 4620 — 300 rpm, 15 sec; 1000 rpm, 20 sec

Cure in oven @ 37°C for 60 min

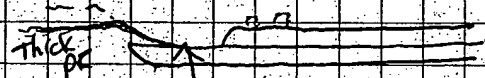
Expose 50 sec

Develop 40 ml 4620 / 100 ml H2O

Results: problem!

22 July 98 — John O'Brien

ie.



Solution(s):
- Try Ashing @ 100 W O2 RIE
- Change PR thickness to 10 μm
  - spin @ 1500 rpm
- Maybe try thin PR just to see what happens
- offset mask for top-shell (compensation)

22 July 98

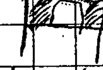
~~DK~~ prepared six more wafers for <u>trials.</u> (A-F)

~~Clean~~ for piranha-etch 5 min
DI rinse 2 min
HF dip 30 sec
DI rinse 2 min
spin dry
deposit Si_3N_4 both sides (40 min)
Sputter Ti/Cu/Cr 3/3/3 min

Trials:

Wafer A: 10 µm Bottom shell, 20 µm Sacrificial PR, 10 µm Top shell
10 µm process: PR 4620 spin 1500 rpm 20 sec
cure + bake the same

Wafer B: ~~10 µm Botto~~ Same as wafer A but, ~~use~~ ~~chrome~~ not chrome! ~~to encapsulate PR~~ (E-Gun Au instead)
30 µm process (PR 4620 1300 rpm 20 sec) ≈ 35 µm for top shell and etch ~~chrome~~ after Top shell micromold is complete. This should force plating to build-up on sides.



Wafer C: Thin PR bottom shell (plate 5 µm), 20 µm Sacrifical, Thin on Top (plate 5 µm)

Wafer D: 10 µm Bottom, 20 µm Sacrificial, Thin PR Top (plate 5 µm)
offset topshell ~~mask~~

* Wafers E+F may be used for grid fabrication that will be used in the assembly process of the old needle design.

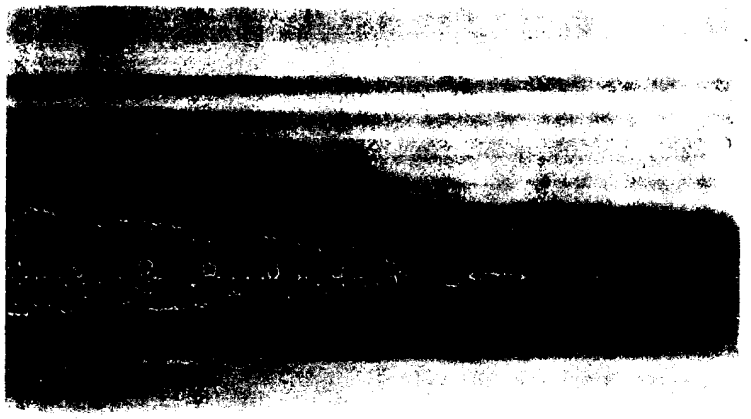
29 July 98

Top shell e-plating

off first two waters (Not trials yet)

plate around

- Strip
- Gold
- Aceto
- DI
- Resu



30 July 98

Trial water "A & B"

10 µm Bottom shell

PR 4620 300 rpm 15 sec
1500 rpm 20 sec
bake 110°C for 2815
expose 40 sec
develop: 400K 40 ml / H_2O 100 ml
Results: very nice

50

29 July 98

Top-shell e-plating

On first two wafers (Not trials yet)

plate around 5 μm @ 15 mA/cm²

- Strip PR
- Gold etch
- Acetone bath
- DI Rinse & dry

Results: - Both wafers withstood high water flow without loss of adhesion.

- Microscope reveals fair appearance.
- Ready for SEM (Later)

30 July 98

Trial wafer "A+B"

10 μm Bottom shell

PR 4620 300 rpm 15 sec
1500 rpm 20 sec
bake 110C for 2:15
expose 40 sec
develop: 400K 40 ml H₂O 100 ml
Results: Very nice

31 July 98

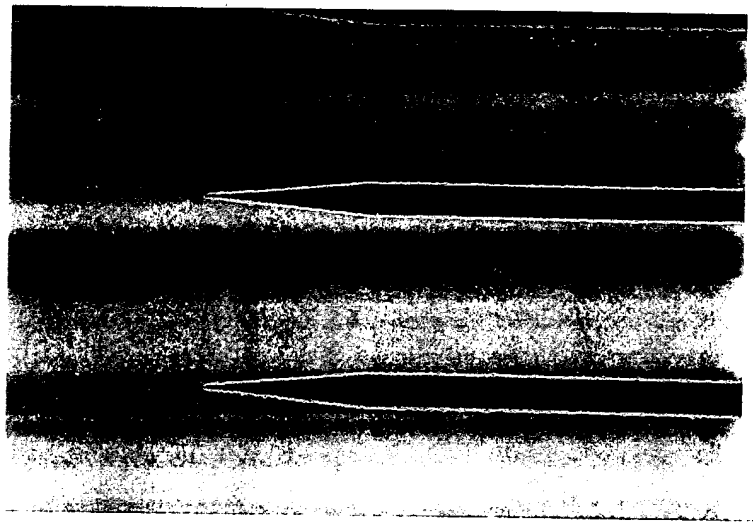
etch Cr in 25% HCl

plate Ni @ 36mA (15mA/cm²) for 35 min

plate Pd @ 36mA for 5 min

Results: very nice

Strip PR in Acetone + Methanol



Bottom Shell ≈10mm

3 Aug 98

Thick PR on bottom shell (Trials A+B)

need ≥ 20 μm

PR 4620 300 rpm 15 sec
1000 rpm 20 sec

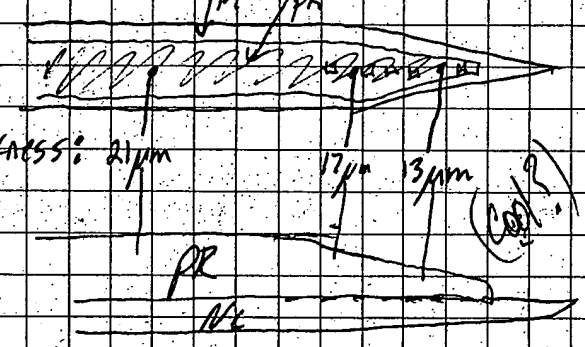
bake 110°C 2:15 min

expose 90 sec

develop 400K (4 min) / H_2O (2 min)

results: Very nice

*note: on wafer A, development of PR down to chrome layer was not allowed. development down to nickel bottomshell was targeted. Also, Z measurement on hi-res microscope revealed following geometry:



5 Aug 98

@ll Trial wafer A:

Sputter Au @ 100W for 3 min ~ 1000 Å
(wafer on heat-sink)

Results: Nice alignment, PR is wrinkled (try slow sputter next time)

" " wafer B:

E-gun Au for 1000 Å

Results: Alignment a little off, PR looks nice

Top shell micromold 10 Aug 98

@ll Trial wafer A:

PR 4620 ~~300~~ 500 rpm 15 sec
~~1500~~ 800 rpm 20 sec ~ 10 μm

bake @ 85°C for 1 hour

expose 50 sec

develop 40 ml 400K/100 ml H_2O

Results: Good!

Wafer B: PR 4620 300 rpm 15 sec
800 rpm 20 sec ~ 27 μm

bake 85°C for 1.5 hours

expose: 65 sec

develop: 40 ml 400K/100 ml H_2O

Results: Good!

19 Aug 98

Shielding

E-plate Top Shell

Wafer Be (22 μm micromold)

Ni bath @ 15mA/cm²

35mA for 2 hours ≈ 34 μm

Results: ports covered-up

Wafer Ti: 35mA for 35 min

Results: Not good

10 Nov 98

New Batch!

12 wafers prep'd and coated w/ oxide & Ti/Cu/Cr

Ver 3

Bottom Shell ≈ 10μm

PR 4620 300 rpm 15 sec
1500 rpm 20 sec

bake 110°C for ~~30~~ 15 min

expose 40 sec

develop 400K 40ml/H_2O 100ml

Results: GOOD!

plate: etch Cr in 75% HCl

Ni @ 35mA (15mA/cm²) for 35 min

Pd @ 12.5mA (5mA/cm²) for 10 min

strip PR + rinse

Results: GOOD

12 Nov 98

Sacrificial layer [add 10 min ash] in O_2 (Branson)

PR 4620 300 rpm 15 sec
1000 rpm 20 sec ≈ 21.7μm

bake 110C 2 min

expose 50 sec

develop 400K 40ml/H_2O 100ml

Results:

Sputter Au @ 50 W for 6 min ← 1000Å
(wafer on heat sink)

Results: wires in PR (encapsulated)

13 NOV 98

Top shell

PR 4620 300 rpm 15 sec
1500 rpm 20 sec

Bake @ 40°C in oven for 1 hour
Expose : 50 sec & add 5 sec
develop 40 ml 400K / 80 ml H_2O
Results: GOOD w/exception of alignment

E plate:
two waters in Ni @ 38 mA (5 mA/cm²) for 35 min
Strip PR under developed
Results: ~~GOOD~~ ~~Adhesion~~

All water in Pd @ 12.5 mA (5 mA/cm²) for 2 hours
Strip PR
Results: GOOD Adhesion?

Lift-Off

~~Gold etch × 30 sec~~
~~5 min Acetone~~
~~2 min Methanol~~
~~Cr etch in HCl ≥ 2 min~~
~~Cu etch ≥ 2 hours in (CuCl₂)~~

~~Results:~~

SEM TIME:

13 Nov 98

Start new Batch using preceding changes! Do on Monday

17 Nov 98

Also add placement of water into ultrasonic water bath prior to top-shell e-plate.

Another Batch

Use process on pgs 55-56 unless otherwise noted.

Bottom shell = 10 μm good!

Sacrificial layer w/ 5 min 300 W O2 RF added

Sputter charge to 1 min ON and 5 min OFF and cycled three times.

Results: Alignment problems! ⟹

24 Nov 98

Top Shell add 5 sec + overlap longer

Results: looks pretty good still have alignment problem e-plate Results: Good! but missing tip of hollow portion of needle

Note: stripping top shell PR to redo for 2 1/2 μm thick specs.

58

17 Dec 98

Results on Thick Top Shell PR (2.1)

Process:

PR4620 300 rpm 15 sec

1000 rpm 20 sec

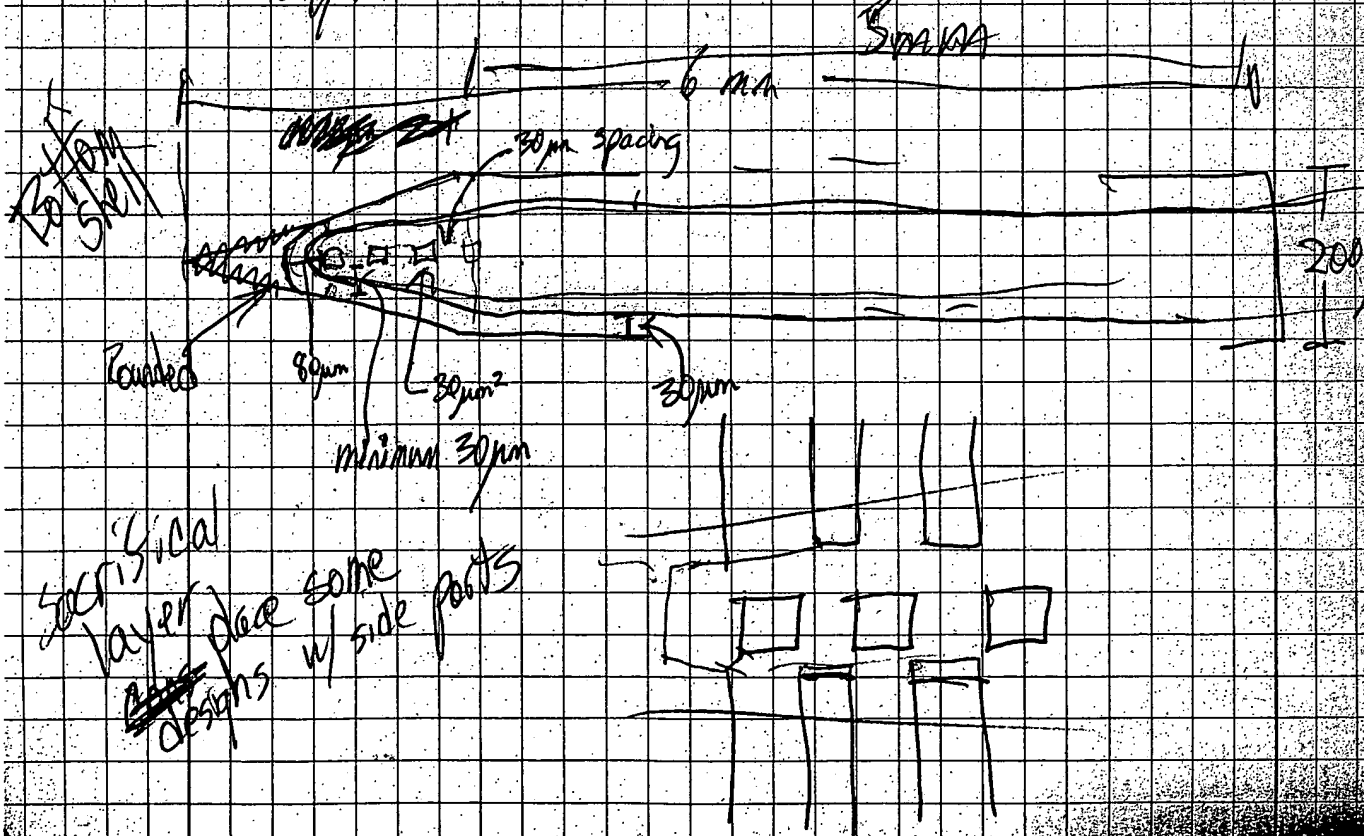
Bake in Oven 50°C 1 hour

Exposed 60 sec

18 Dec 98

Redesign Mask!

Length: Same

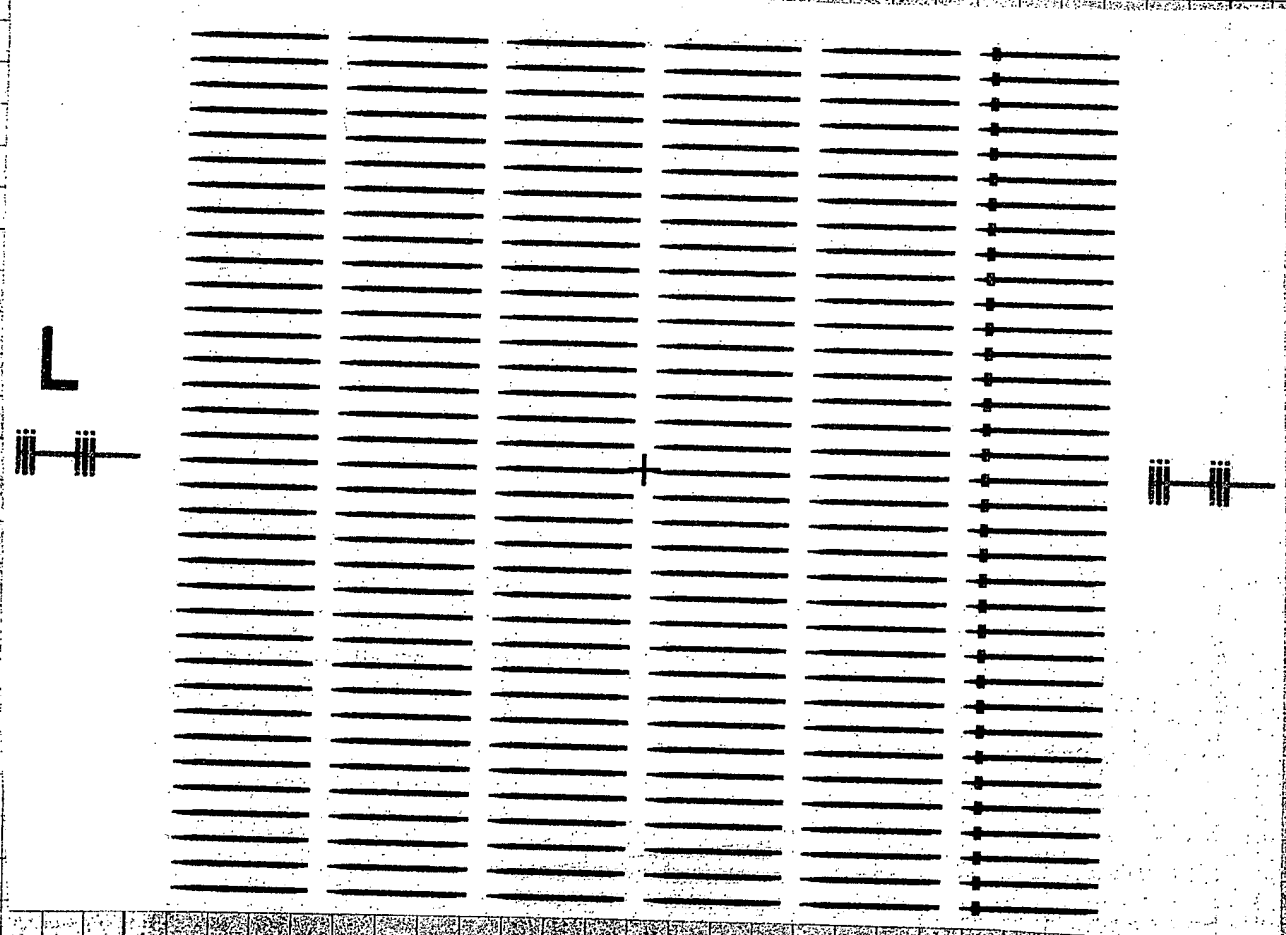


Typical layer designs w/ side posts

Top Shell

Sun

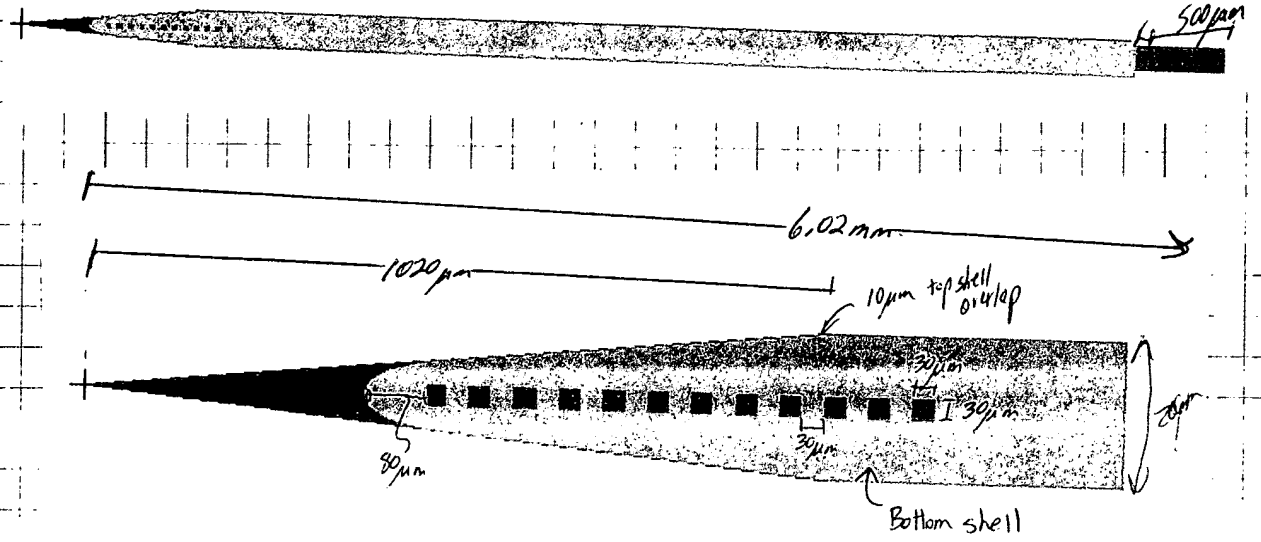
6 Jan 98 Completed Mask Design

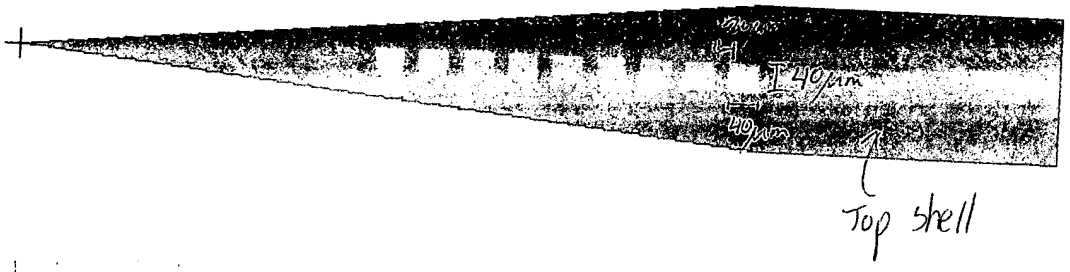


Layer 46 Underside
Layer 49 Top shell
Layer 43 Bottom shell

This column contains side port design

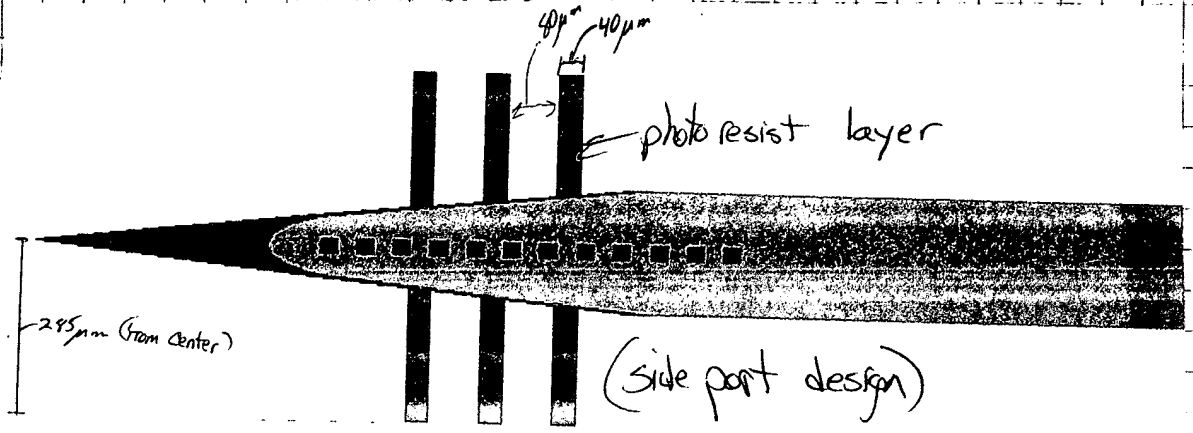
60 11 Jan 99

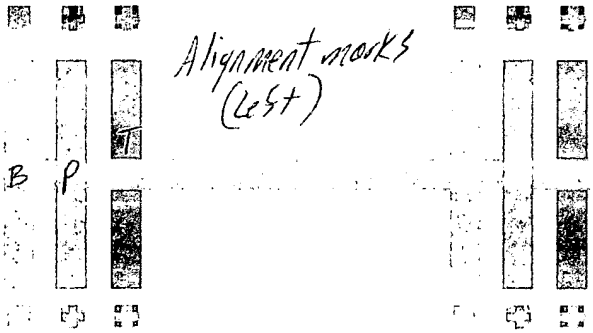

500 μm
6.02 mm
1020 μm
10 μm top shell overlap
30 μm
30 μm
30 μm
80 μm
30 μm
Bottom shell


40 μm
40 μm
Top shell

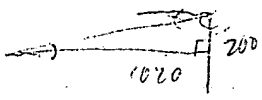

140 μm
60 μm
20 μm
photo resist layer

11 Jun 98





Alignment marks (test)



20 Jan 99

All masks in hand!

Clean wafers w/ Ti/Cu/Cr

Bottom shell ≈ 10μm

PR 4620 300 rpm 15 sec
1500 rpm 20 sec ≈ 12μm

bake 110C hot plate for 2:15

expose: 40 sec

develop: 400K 400ml H_2O 100ml

Results: Good!

e-plate

Ni @ 37mA for 35 min ≈ 10μm

Au @ 12.5mA for 10 min ≈ 1μm

Strip PR w/ acetone + methanol

Results: peel-off!

Edges poor Cu later or need to stay longer in HCl!

21 Jan 99

Sacrificial Layer

PR 4620 300 rpm 15 sec
1000 rpm 20 sec ≈ 2.4 μm
bake 110°C for 2:15
expose 50 sec
develop 400K
Results: Not Bad
~~Watch~~ watch for overdeveloping

e-gun Au ≈ 1000 Å
* problem w/ pressure during evaporation.

Top Shell

21 Jan 99

PR 4620 300 rpm 15 sec ≈ 2.4 μm
1000 rpm 20 sec
Bake in Oven @ 50°C 1 hour
expose: 60 sec + develop 400K
Results: GOOD

note: PR of sacrificial layer
is being etched.

e-plate Ni @ 3mA for 1 hour
* plating on top of Sac layer occured immediately.
Results: looks pretty good, need SEM to verify side-walls.

29 Jan 99

Started another Rev

- pattern bottom shell
- e-plate ≈ 10mm

Results: very nice! use 1/4 second ultrasonic bath prep to plating

4 Feb 99

- Sacrificial layer followed by 5 min ash

Results: GOOD appearance from top

- Sputter Au: 50W for 1 min
  w/ 5 min cool
  (3 cycles)

Results: Very nice!
No waves.

5 Feb 99

Top shell
PR and bake
expose 60 sec + develop

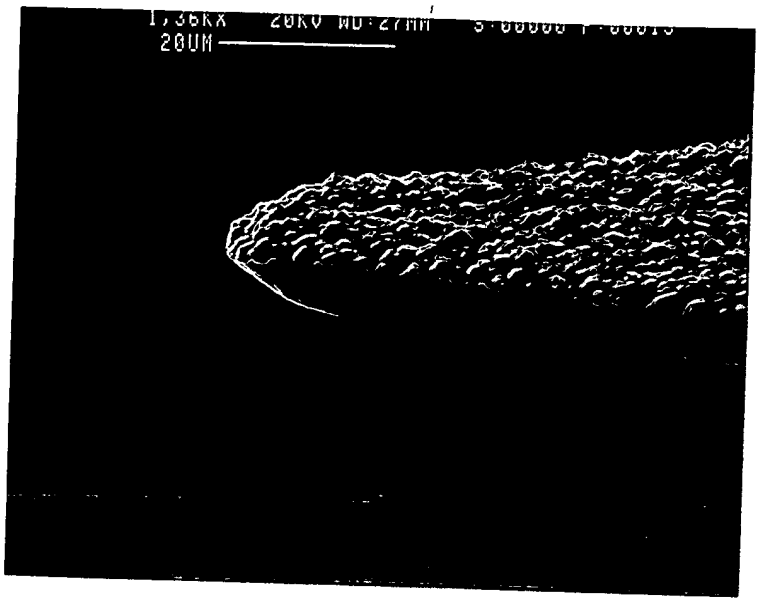
Results: GOOD.

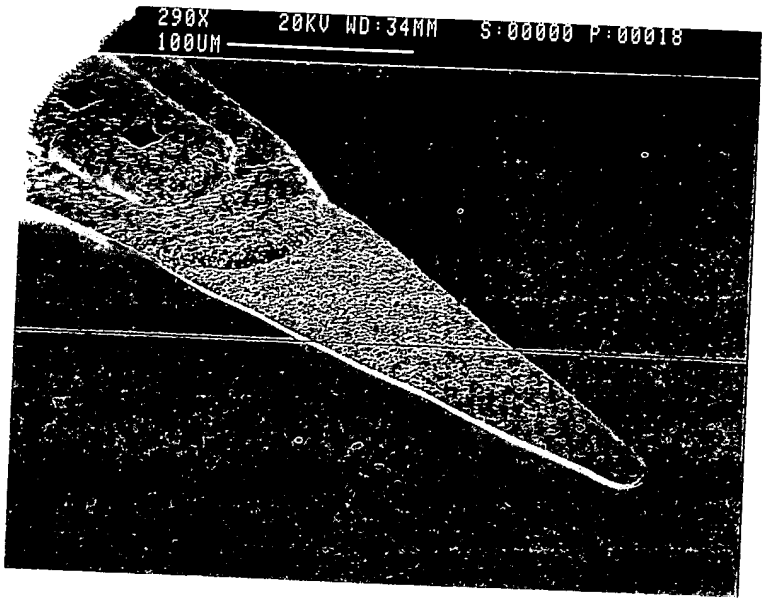
6 Feb 99

e-plate Ni @ 37 mA for 1 hour

Results: GOOD.

SEM's Looks pretty cool

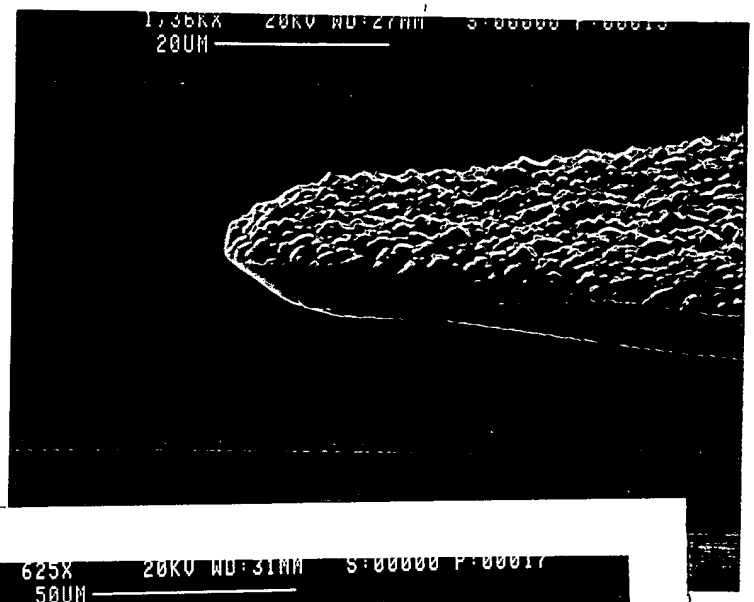


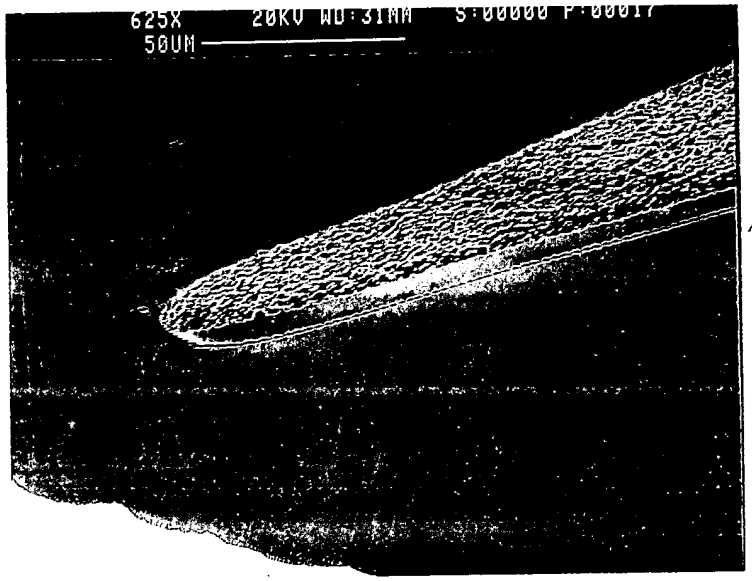


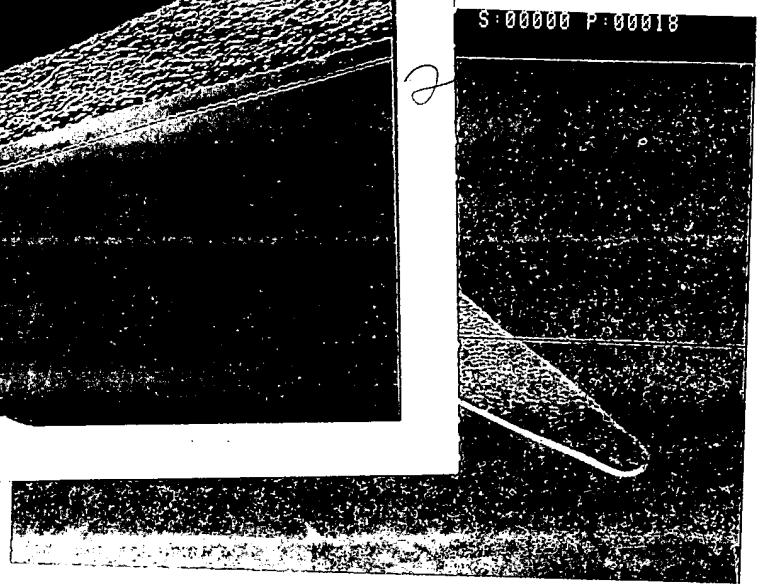
Polaroid MG2724A704186

11 Feb 99

SEM's Looks pretty good



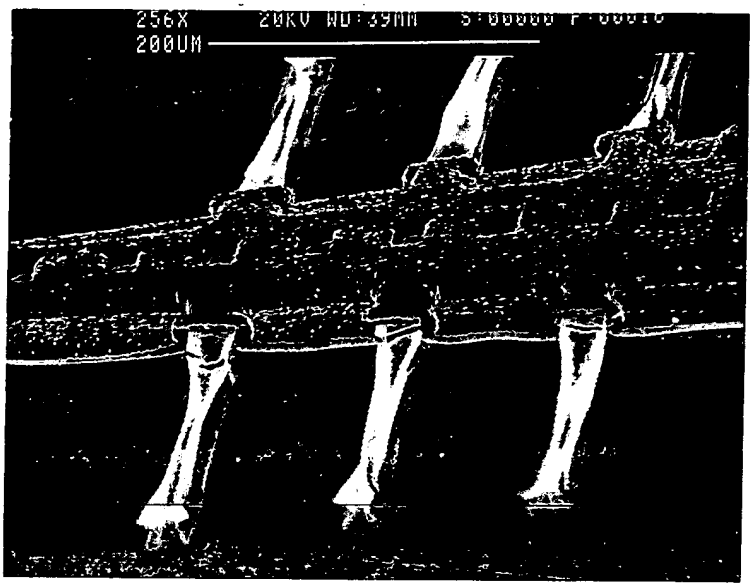


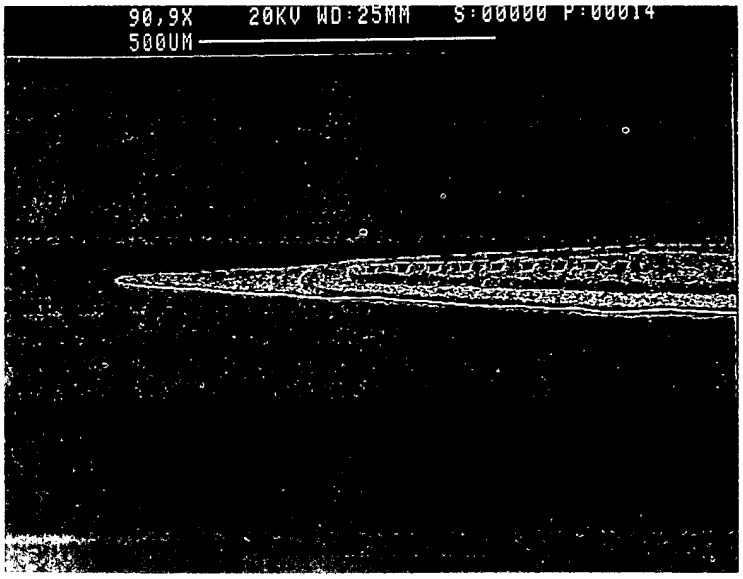


66

11 Feb 99

SEM

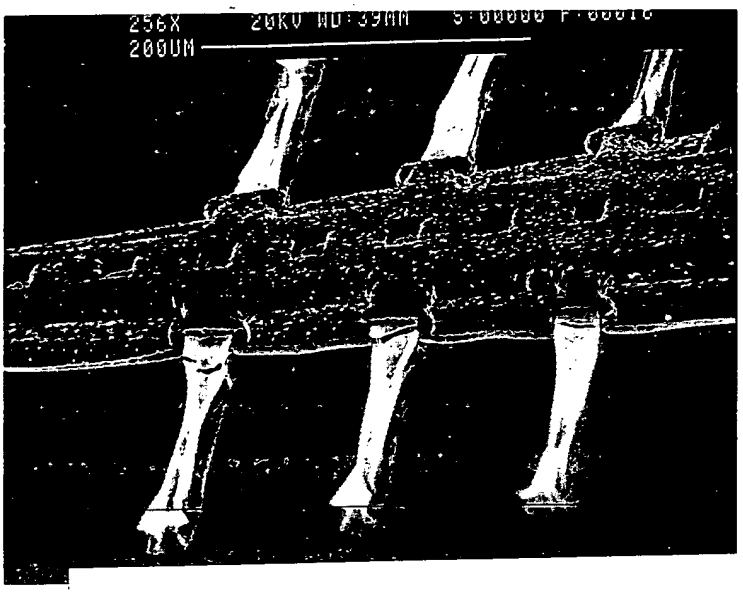


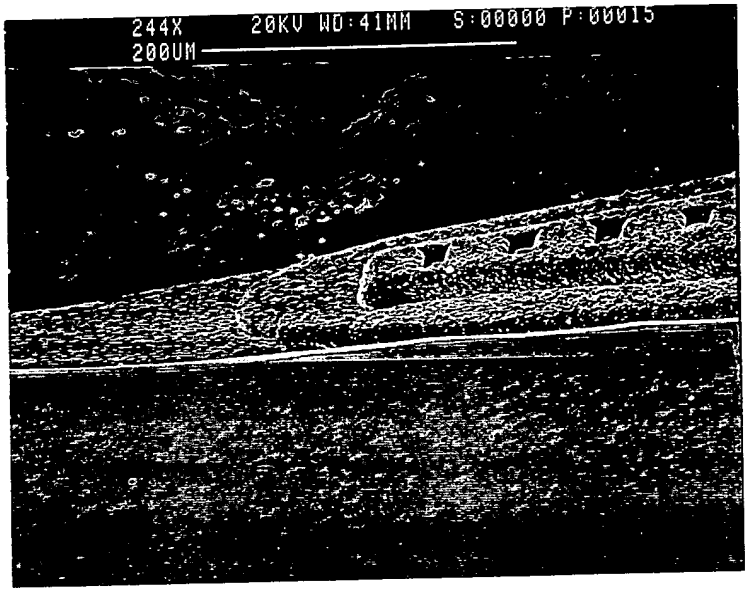


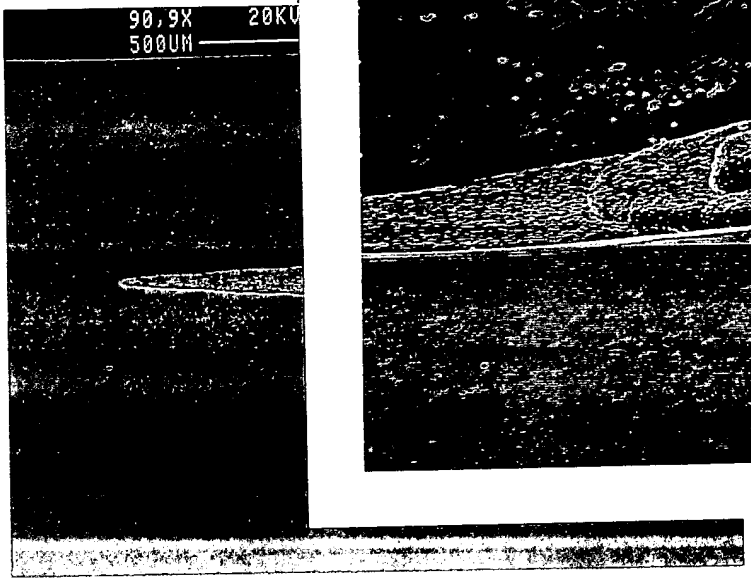
66

11 Feb 99

SEM







11 Feb 97

Release Needles

1- 1 sec in ultrasonic bath w/ DI water
2- 15 sec Au-etch
3- 10 min Acetone
4- 5 min 2-propanol
5- Cr-etch 1 min
6- Ammonium Hydroxide saturated w/ copper sulphate
≈ 1.5 hours

Results: Good!

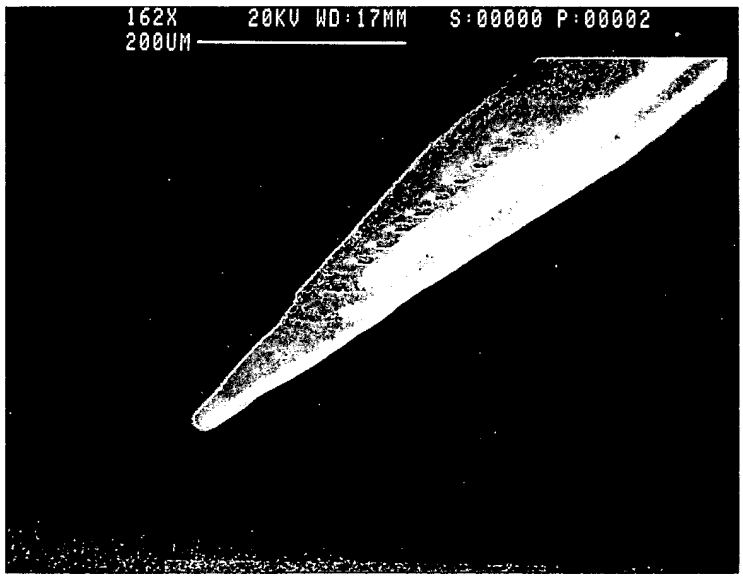
* End up using the yellow Cr-etch. Surprisingly it removed the Cu layer in under a minute without damaging the nickel.

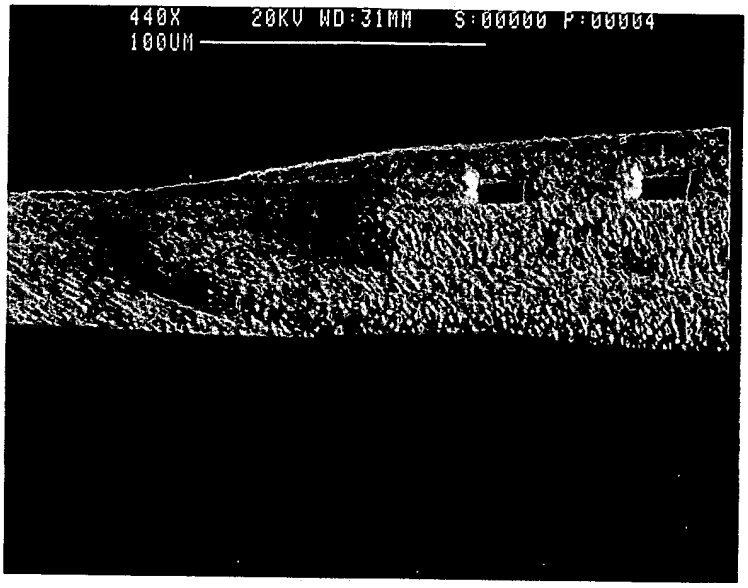
17 Feb 99

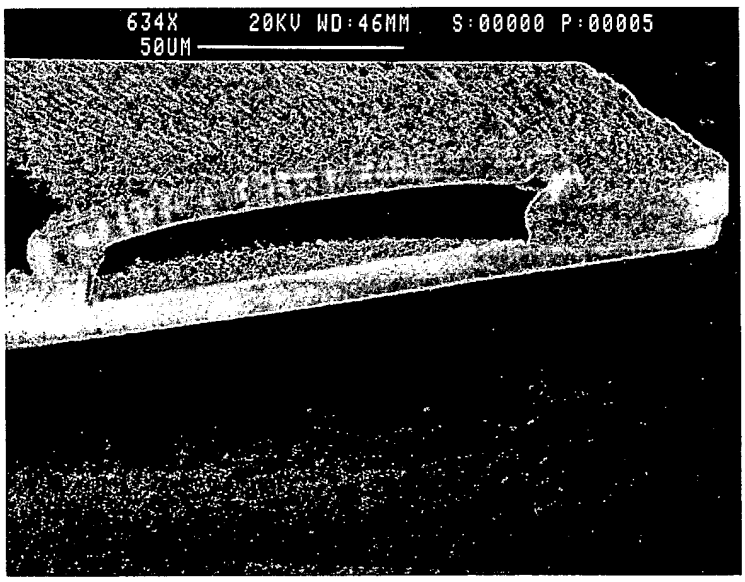
Released the nicer of the two wafers + mounted for SEM.

23 Feb 99

Results Good!



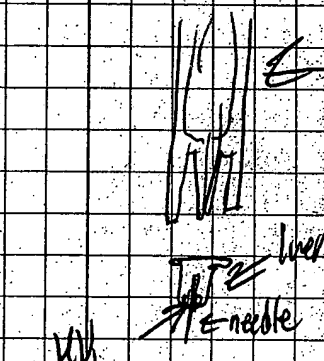

440X 20KV WD:31MM S:00000 P:00004
100UM


634X 20KV WD:46MM S:00000 P:00005
50UM

1 Mar 99

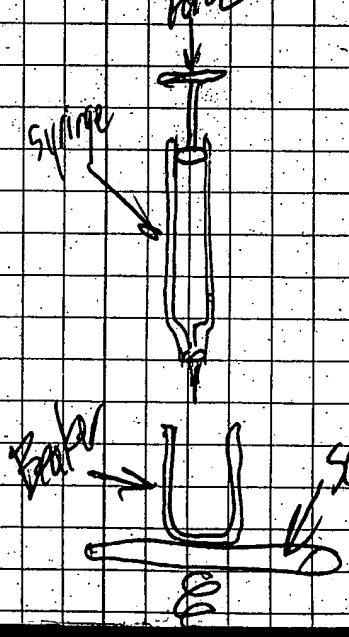
Attempts to thicken shell. I can go 20 μm as thick as I can go without losing parts.

3 Mar 99 packaging



need pictures!

11 Mar 99 Testing



* Apply constant force over period of time and measure mass of water.

14 Mar 99

Preliminary testing results

Max: -10.955 cycle speed: 10 mm/min
Min: -10 lbs

Run 30 min ⇒ Mass$_{60}$: 1.3952 g

* Experiment w/ speed to get best constant force.

16 Mar 99

Needle testing

Max: -9.975 cycle speed: 10 mm/min
Min: -10.000 5

@ ~~250~~ 30 minute runs: Needle #

1 - .7194 g
2 - .6713 g
3 - .8172
4 - .9458
5 - .7606
6 - .7487
7 - .7615

17 Mar 99

Testing From yesterday

8 - .7578

9 - .7364

10 - .7563

at 20 lbs force needle ⓪

max: 19.97 min: 20

1 - 2.3304 g

2 - 2.6946

3 - 2.8303 Chg to 15 min run

23 Mar 99 Needle ⓪

30 Mar 99

Force → pressure conversion d = .485" by micrometer

Area of syringe plunger: $\frac{\pi d^2}{4} = \frac{\pi (.485)^2}{4} = .1847 in^2$

pressure scale

For 100 psi → F = 18.47 lb

90	16.623	40	7.388	1	.1847
80	14.776	30	5.541	.5	.09235
70	12.929	20	3.694	.1	.01847
60	11.082	10	1.847		
50	9.235	5	.9235		
		4	.7388		
		3	.5541		
		2	.3694		

30 Mar 99

min: -1.005
max: -.9995
speed: 10 for 30 minutes

1 lb F ⇒ > 5 psi

Needle IV

1- .0035
2- .0060
3- .0053
4- .0065

1 psi min: -1.850 for 30 minutes
max: -1.845
speed: 5

Needle IV

1- .0052
2- .0058

gravity for 30 minutes

1- .0040
2- .0046

10 psi min: -1.850 speed: 10 30 min
max: -1.845

31 Mar 99

1- .0064
2- .0073

20 psi MIN: -3.700 speed: 10 30 min
max: -3.690

1- .0475 3- .0404 5. 0279
2- .0606 4- .0381 6. 0194

6 14 Apr 99

30 psi min: -5.545
max: -5.535

speed 2/60 30 min

1- .1290
2- .1156
3- .1230
4- .1217

Flow calculation

$m = dV$

$d_{H_2O} = 1\ g/cm^3$ @ room Temp

$\therefore V = \frac{.1230\ g}{1\ g/cm^3} = .1230\ cm^3$

Rate = .0041 cc/min = .2460 cc/sec

13 Apr 99 All data previous is wrong

30 psi min: 5.543
max: -5.535

speed 1.5 30 min

1- 4.1514 g

evap rate: 4.1501 over 30 min
4.1485

2- Spring leak

1 psi min: -.1850
max: -.1845

speed 2 30 min

1- .0035
2- .0033
3- .0074
4- .0056
5- .0060
6- .0060
7- .0055
8- .0067

Needle st

$\Delta w = \pm .0001$ | $w =$

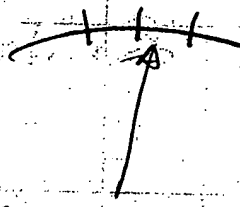
$\Delta \tilde{F} = \pm .5\%$ | $F =$

$\Delta \tilde{d} = \pm .0005"$ | $d = .485$

$\Delta \tilde{t} = .115$ | $t = 30\ min$
$= .001667\ min$

$m_{final} =$ | $\Delta \tilde{m}_{final} \approx .0001$

$m_{evap} =$ | $\Delta \tilde{m}_{evap} \approx .0001$



$$P = \frac{F}{A} = \frac{F}{\pi d^2/4}$$

$$\Delta P = \left|\frac{\partial P}{\partial F}\right| \Delta \tilde{F} + \left|\frac{\partial P}{\partial d}\right| \Delta \tilde{d}$$

↑ sensitivity coefficients

$$= \left|\frac{1}{\pi d^2/4}\right| \Delta \tilde{F} + \left|\frac{2F}{\pi/4\, d^3}\right| \Delta \tilde{d}$$

$$= \left|\frac{1}{\pi d^2/4}\right| \Delta \tilde{F} + \frac{2F}{\pi d^3/4} \Delta \tilde{d}$$

$$\Delta P = \frac{1}{\pi(.485)^2/4}(F)(.005) + \frac{2F}{\pi(.485)^3/4}(.0005)$$

.151 high
.014 low
$\Delta P = .0825$

$P = 20 \pm .05\ psi$

$$Q = \frac{m_{final} + m_{evap}}{t}$$

$$\Delta Q = \left|\frac{\partial Q}{\partial m_f}\right| \Delta \tilde{m}_f + \left|\frac{\partial Q}{\partial m_{evap}}\right| \Delta \tilde{m}_{evap} + \left|\frac{\partial Q}{\partial t}\right| \Delta \tilde{t}$$

$\frac{1}{t}$ × .0001 | .0001 | $\frac{m+m}{t^2}$ × .001667

based on instrument reading
actual uncertainty may be higher

High 1.55×10^{-5}
Low 6.68×10^{-6}
$\Delta Q = .000011$

12 May 99

1psi Min: -.1850 Speed: .2 for 30 min
Max: -.1845

Runs: 1. .0215 (flow) 6. .0051
2. .0044 7. .0056
3. .0049 8. .0058
4. .0051 9. .0064
5. .0049 10. .0052

2psi Min: ~~-.3700~~ -.3700 Speed .2 for 30 min
Max: -.3690

Runs: 1- .0044
2- .0060

13 May 99

3- .0059
4- .0054
5- .0050

3psi Min: -.5545 Speed .2 @ 30 min
Max: -.5535

1- .0050 (same as previous)

4psi → Min: -.9240
Max: -.9230

1-

17 May 99

Needle VI

3 psi Min: -.9240 @ .2 for 30 min
Max: -.9230

1- .0134
2- .0086
3- .0111
4- .0098
5- .0091
6- .0089
7- .0100
8- .0097
9- .0090

---

18 May 99

10 psi Min: -1.855 @ .2 for 30 min
Max: -1.840

1- .1332
2- .1140
3- .0980
4- .0739
5- .0727
6- .0761
7- .0707
8- .0673
9- .0656
10- .0416

20 June 99

10 psi min: −1.855 @ .2 for 30 min
max: −1.840

1- .0483 6- .0343
2- .0687
3- .0644
4- .0376
5- .0321

18 June 99
10 psi again
1- .1454

---

21 June 99
2- .1137 5-
3- .0481
4- .0292

20 psi min: −3.07 0.2 for 30 min
max: −3.69

1- .07358

~~June~~ 99 4- 1.1587 7- 1.1787
2- 1.2024 5- 1.0999
3- 1.1225 6- .9635

29 June 99

30 psi -5.545 = min
-5.535 = max 0.2 for 30 min

1- 1.6928
2- 1.6215
3- 1.7028
4- 1.6135
5- 1.6842

July 99

40 psi -7.390 = min
-7.380 = max 0.2 for 30 min

1- 2.2054
2- 2.3402
3- 2.1089
4- 2.2531
5- 2.4018
6-

50 psi -9.230 = max .2 for 30 min
-9.2410 = min

1- 3.0630
2- 3.1811
3- 3.2218
4- 3.0528
5- 3.1554
6-

60 psi -11.08 = max .2 30 min
-11.09 = min

1- 4.1964
2- 3.9776
3- 4.2154
4- 4.0345
5- 4.1332
6-

70 psi -12.935 = min 0.2 for 15 min
-12.985 = max

1- 2.4535
2- 2.3566

19 July 99

Goal: Make needles thicker!

Clean wafers w/ Ti/Cu/Cr

Bottom shell ≈ 17μm of Ni

-PR 4620 3000 rpm 15 sec
1000 rpm 20 sec
bake 110°C for 2:15 (yields 21μm mold)
expose: 110 sec
develop: 400K (40ml) / H_2O (100ml)
Results: mostly slightly overexposed?
(.5 sec ultrasonic bath prior to Cr etch)
- etch Cr in 25% HCl
Ni @ 57mA for 1 hour ≈ 18μm
Pd @ 125mA for 10 min ≈ 1μm
strip PR w/ acetone + methanol
Results: good but output ports are covered

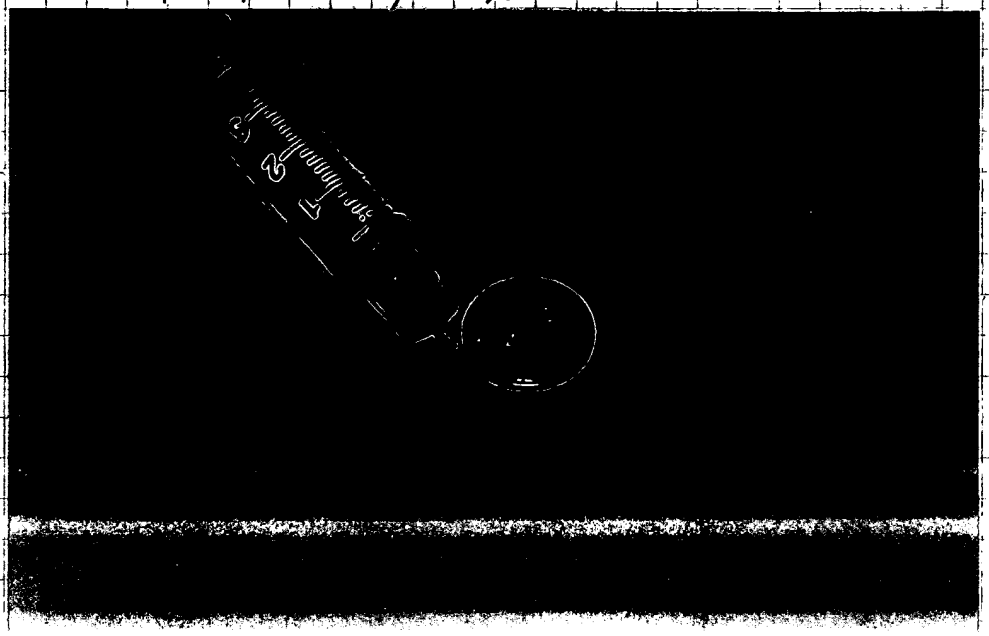
1 Aug 99

Sacrificial layer

PR 4620 300 rpm 15 s
1000 rpm 20 s
bake: 110°C for 2:15
expose: 10 sec
develop: 400K 40ml / H_2O 100ml
Results:

pictures of packaged needle

15 Sep 99



15 Apr 99

travel grant, and (6) proposed budget.

THE UNIVERSITY OF UTAH

Micro Instrumentation Research Laboratory

Department of Bioengineering

50 S. Central Campus Drive
Room 2480
Salt Lake City, Utah 84112

John D. Brazzle
Microneedle Research and Bioinstrumentation Systems
Tel: (801) 581-8611
E-mail: brazzle@eng.utah.edu
Web: www.eng.utah.edu/~brazzle